TOSCA User Manual

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PREFACE

This document is designed as an *aide-memoire* to help you run your experiment and analyse your data. **It is not intended as a substitute for training!** For new users and those who are not regular users, it is essential that you are properly trained in the use of the instrument by your local contact or the instrument scientist.

More detailed information on some aspects is available from other reports; such as the sample environment equipment, FRILLS (a fit to a sum of Gaussian peaks) and on programs such as GENIE. Copies of these manuals can be obtained from your local contact, although copies are kept in the instrument cabin. A PUNCH manual can be found in the cabin and contains information on the Instrument Control Program (ICP) and sample environment controls via CAMAC.

This manual is specific for current version of TOSCA, a pdf file is also available for download.

TOSCA
CLRC
KENT

Team TOSCA: left to right, Timmy Ramirez-Cuesta, Stewart Parker (Instrument Scientists) and John Tomkinson (Group Leader, Molecular Spectroscopy Group).

1 TOSCA

1.1 Description of the instrument and basic INS theory

TOSCA is an inelastic neutron scattering (INS) spectrometer optimised for vibrational spectroscopy. TOSCA has replaced the previous spectrometers TFXA and TOSCA-1, however, it retains the advantages of the earlier instruments (ease of operation and reliability) but simultaneously offers improved sensitivity and resolution.

TOSCA is a collaborative project between the Consiglio Nationale Recherche (CNR) of Italy, the Department of Physics at the University of Kent at Canterbury (UK) and ISIS. (For a detailed list of the participants see the TOSCA website http://www.isis.rl.ac.uk/molecularspectroscopy/tosca/ and the CNR website: http://www.ifac.cnr.it/tosca/tosca-main.htm). TOSCA was installed in two phases: phase 1 was completed in May 1998 and consisted of the backscattering spectrometer at 12 m, phase 2 added detectors in forward scattering and moved the spectrometer to 17.0 m. This was completed in September 2000. The move to 17 m from 12 m has resulted in a large improvement in resolution.

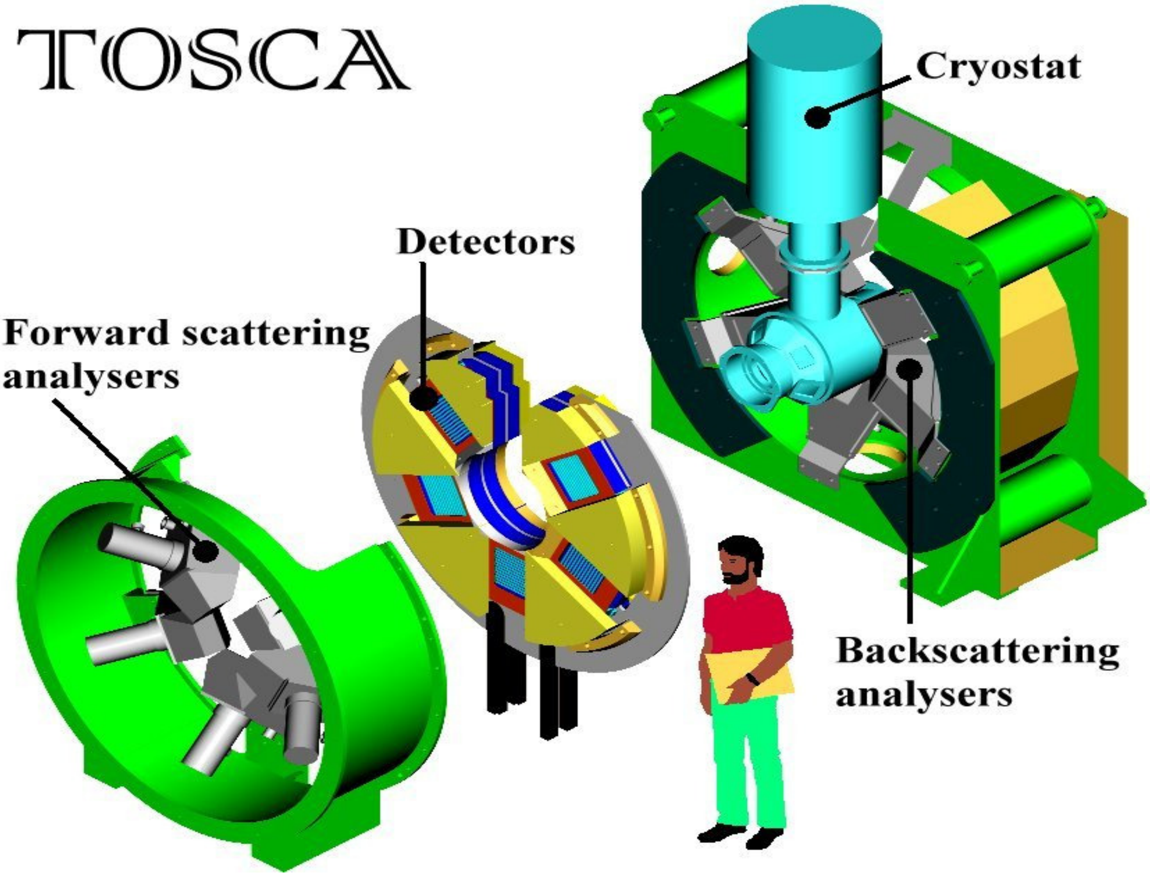

TOSCA
Cryostat
Detectors
Forward scattering analysers
Backscattering analysers

Figure 1.1. TOSCA showing the detector/analyser modules.

TOSCA is an indirect geometry time-of-flight spectrometer at the ISIS pulsed spallation neutron source at the Rutherford Appleton Laboratory. A section through one of the analyser modules is shown in *Figure 1.2.* It is optimal in the energy range 0 - 4000 cm^{-1} (0 - 500 meV) with the best results below 2,000 cm^{-1}, (250 meV). To suppress the &gamma-flash and fast neutron background a Nimonic chopper is installed at 9.5 m. This has a tailcutter (a sheet of B_4C) on the leading edge to remove low energy neutrons that would otherwise result in frame overlap.

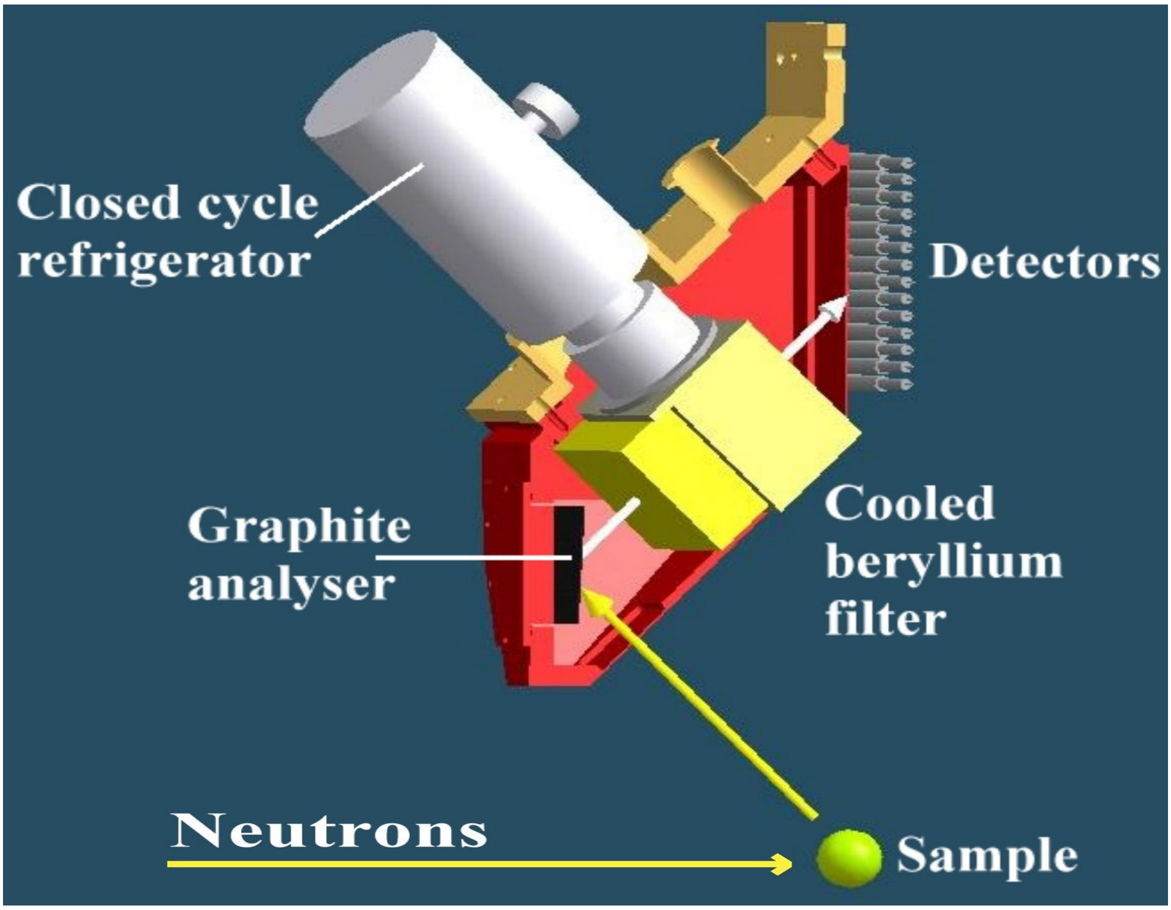


Figure 1.2. A section through one of the analyser modules of TOSCA.

The source of neutrons on TOSCA is the white beam from the water moderator. The time-of-flight technique is used for energy analysis of the scattered neutrons. A small fraction of the incident neutrons are inelastically scattered by the sample; those that are backscattered through an angle of 45° or 135° impinge on a graphite crystal. Bragg's law states:

$$\lambda = 2\, d \sin\theta \qquad (1)$$

where d (Å) is the interplanar distance in the crystal, λ (Å) is the wavelength of the scattered neutron and θ is the angle of incidence on the crystal.

From equation (1), since both d and θ are constant only one wavelength (and its higher orders, λ/2, λ/3 etc...) will be Bragg scattered by the crystal, the remainder will pass through the graphite crystal to be absorbed by the shielding. The neutrons at multiples of the fundamental wavelength are scattered by the beryllium filter which acts as a longpass filter and the remaining neutrons are then detected by the ^{3}He filled detector tubes. The net effect of the combination of the graphite crystal and beryllium filter is to act as a narrow bandpass filter.

The energy transferred to the sample, E_{trans}, is:

$$E_{trans.} = E_{i.} - E_f \qquad (2)$$

where E_i and E_f, are the incident and final energies respectively. The kinetic energy, E, of a neutron is given by:

$$E = \frac{mv^2}{2} \qquad (3)$$

where m is the mass of the neutron and v is its velocity. Rearranging (3) gives:

$$v = \sqrt{\frac{2E}{m}} \qquad (4)$$

and since

travel time = distance / velocity (5)

It follows that the time of arrival at the detector, T, is the sum of the time from the moderator to the sample, t_i, and the time around the analyser, t_f, thus:

$$\begin{aligned} T &= t_i + t_f \\ &= \frac{L}{v_i} + \frac{l}{v_f} \\ &= L\Big/\sqrt{\frac{2E_i}{m}} + l\Big/\sqrt{\frac{2E_f}{m}} \end{aligned} \qquad (6)$$

Now since the final energy, E_f, the distance round the analyser system, l, and the length of the flight path from the moderator to the sample, L, are all known, it follows that the time of arrival at the detector uniquely defines the incident energy, $E_{i.}$ and hence the energy transfer at the sample, E_{trans}. Thus it is a simple matter to convert from time-of-flight to energy. The result is a spectrometer with no moving parts than can record spectra from 0 to 8000 cm^{-1}, although the best results are usually obtained below 2000 cm^{-1}. The resolution of the spectrometer is determined by a number of factors but for practical purposes can be taken to be 1.25% of the energy transfer.

The intensity of the ith molecular vibrational transition is proportional to:

$$I_i \propto Q^2 U_i^2 \exp\left(-Q^2 U_{Total}^2\right) \sigma \qquad (7)$$

Since neutrons have a mass approximately equal to that of the hydrogen atom, an inelastic collision results in a significant transfer of momentum, Q ($Å^{-1}$), as well as energy, to the molecule. On TOSCA the design is such that there is only one value of Q for each energy, ($Q^2 \approx E_{Trans}/16$). (Other instruments at the ISIS Facility and the ILL allow both the energy and the momentum transfer to be varied, but they constitute a different story). U_i is the amplitude of vibration of the atoms undergoing the particular mode. The exponential term in equation (7) is known as the Debye-Waller factor, U_{Total} is the mean square displacement of the molecule and its magnitude is in part determined by the thermal motion of the molecule. This can be reduced by cooling the sample and so spectra are typically recorded below 50K.

σ is the inelastic neutron scattering cross-section of all the atoms involved in the mode. The scattering cross-sections are a characteristic of each element and do not depend on the chemical environment. The cross-section for hydrogen is ~ 80 barns while that for virtually all other elements is less than 5 barns. This means that modes that involve significant hydrogen displacement will dominate the spectrum. This dependence on the cross section is why the INS spectrum is so different from infrared and Raman spectroscopies. There, the intensity derives from changes in the electronic properties of the molecule that occur as the vibration is executed, (the dipole moment and the polarisability for infrared and Raman spectroscopy respectively).

Analysis of INS spectra can be carried out in many ways. For molecular systems, normal coordinate analysis using the Wilson GF matrix method as implemented in CLIMAX has long been used. More recently, ab initio methods have proven to be very successful. The programme aCLIMAX uses the atomic displacements generated by ab initio packages such as GAUSSIAN98 and DMOL3 to generate the INS spectrum. See Further Reading at the end of this section for more details on CLIMAX and aCLIMAX.

A consequence of the indirect geometry is that for energy transfers >100 cm^{-1} the momentum transfer vector is essentially parallel to the incident beam. The significance is that for an INS transition to be observable there must be a component of motion parallel to the momentum transfer vector. This means that with oriented samples (such as single crystals or aligned polymers) measurements directly analogous to optical polarisation experiments carried out .

In addition to the inelastic detectors there are also two ^{3}He filled detector tubes either side of the incident beam (scattering angle ≈ 179º). These are for elastically scattered neutrons and enable modest resolution, $\Delta d/d \approx 3 \times 10^{-3}$, diffraction patterns to be recorded simultaneously with the inelastic spectrum. It is planned to install two further banks of diffraction detectors at 45 and 135º. The purpose of the detectors is to provide a check on the crystal phase of the material and to monitor phase changes as an experimental variable is changed *e.g.* temperature and pressure. There is also a low efficiency scintillation detector (the monitor) in the main beam just before the cryostat vacuum tank. This measures the incident flux distribution as a function of time and is used to normalise the spectra.

Further reading

There is more information about TFXA and TOSCA at the Molecular Spectroscopy website: http://www.isis.rl.ac.uk/MolecularSpectroscopy/. This includes a list of publications resulting from work on TFXA and TOSCA. There is also a database of INS spectra that have been obtained on the instruments. Two types of ASCII and two types of image files are available for downloading. The database is at:

http://www.isis.rl.ac.uk/INSdatabase/

D. Colognesi, M. Celli, F. Cilloco, R. J. Newport, S. F. Parker, V. Rossi-Albertini, F. Sacchetti, J. Tomkinson and M. Zoppi.
TOSCA neutron spectrometer; the final configuration,
Appl. Phys. A 74 [Suppl.] (2002) S64-S66.
A description of the current version of TOSCA.

S. F. Parker, C. J. Carlile, T. Pike, J. Tomkinson, R. J. Newport, C. Andreani, F. P. Ricci, F. Sachetti and M. Zoppi,
TOSCA: A World Class Inelastic Neutron Spectrometer
Physica B 241-243 (1998) 154-156.
This paper gives an overview of TOSCA.

S F Parker,
Vibrational Spectroscopy With Neutrons
Spectroscopy Europe 6 (1994) 14-20.
This gives a brief description of TFXA and highlights some of the areas of research on the instrument.

J Tomkinson
The Vibrations of Hydrogen Bonds
Spectrochimica Acta, 48A (1992) 329-348.
Illustrates the application of INS to hydrogen bonding studies.

G J Kearley,
A Review of the Analysis of Molecular Vibrations Using INS
Nuclear Instruments and Methods in Physics Research, 354 (1995) 53-58.
An excellent overview of how to analyse INS data using normal coordinate analysis.

J Tomkinson
Neutron Molecular Spectroscopy
in *Recent Experimental and Computational Advances in Molecular Spectroscopy,*
(R Fausto ed.) Kluwer, 1993 pp229-249.
Briefly describes the theory of INS (and references to in-depth treatments) and some applications.

aCLIMAX is available from http://www.isis.rl.ac.uk/molecularspectroscopy/
It is described in:
D. J. Champion, J. Tomkinson and G. J. Kearley
a-CLIMAX: a new INS analysis tool
Appl. Phys. A 74 [Suppl.] (2002) S1302-S1304
and
A. J. Ramirez-Cuesta

aCLIMAX 4.0, The new version of the software for analysing and interpreting INS spectra
Computer Physics Communications, in press 2003.

B. S. Hudson
Inelastic neutron scattering: a tool in molecular vibrational spectroscopy and a test of ab initio methods
J. Phys. Chem. A, 105 (2001) 3949-3960.
A demonstration of the power of the combination of ab initio calculations and INS spectroscopy.

1.2 Sample Environment on TOSCA

The beam size at the sample position is 40 mm high by 40 mm wide. It is clearly advantageous to fill as much of the beam as possible. For the best resolution the sample should be 1 mm thick, however, samples up to 4 mm thick are usable.

Solid samples are usually just wrapped in aluminium foil and attached to a centrestick (see section 2.2 Preparing samples). Liquid samples are run in thin walled aluminium cans. Air or moisture sensitive samples (solid or liquid) should be loaded into the cans in a glovebox.

As explained in the previous section, to maximise the INS intensity it is necessary to reduce the Debye-Waller factor as much as possible, thus virtually all samples on TOSCA are cooled. Cooling below 50K makes little difference to the spectrum, thus a Closed Cycle Refrigerator (CCR) which attains temperatures below 20K region is adequate for most samples, see *Figure 1.3*. This has the virtues of being reliable, cheap to run and simple to operate. The CCR is isolated from where the sample sits and uses helium exchange gas to cool the sample. This has the advantage that the sample can be changed without warming the CCR, thus samples can be changed in a matter of minutes without difficulty (see section 2.5 Changing a sample).

The TOSCA CCR has a an internal diameter of 100 mm so will take standard ISIS centresticks. In addition, there are a number of dedicated centresticks for special applications. The most important of these is the 24 position automatic samplechanger. This is described in detail in section 2.4 and 3.5.

If temperatures below the base temperature of the CCR (~ 20K) are required then normal ISIS practice would be to replace the CCR with a liquid helium cryostat ("orange cryostat"). Because of space constraints this is not possible on TOSCA. Instead, there is a centrestick that incorporates a liquid helium bin in its shaft. The design is such that it holds sufficient liquid helium to enable a spectrum to be recorded, see *Figure 1.3*. It may also be pumped on to give a base temperature of ~ 1.5K. It is available for use but must be requested well in advance of the experiment (a minimum of two weeks and ideally on the proposal form).

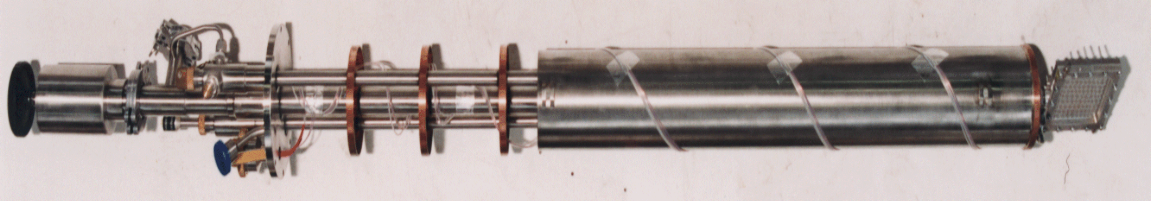


Figure 1.3: The liquid helium centrestick for use on TOSCA

For pressure experiments there are two options. For pressures up to 4 kbar, the helium intensifier should be used. This allows relatively large samples (the can is 7 mm diameter x 40 mm long) to be used and the pressure can be adjusted with the centrestick in the beam. For higher pressures, the McWhan clamped cell is used. The McWhan cell, see *Figure 1.4*, uses pre-stressed alumina inserts to achieve pressures of up to 25 kbar. The sample sizes are of the order of 4 mm in diameter and 10 mm long. It is not possible to pressurise in-situ, and it takes several hours to cool the whole cell once it is on the instrument. **If experiments at pressure are intended then the equipment must be requested on the proposal form. It is not available on demand.**

Figure 1.4: A McWhan cell for pressure experiments up to 25 kbar.

2. SAMPLE HANDLING

This section describes how to prepare a sample, load it into TOSCA and what to do if a centrestick gets stuck in the instrument.

2.1 Safety

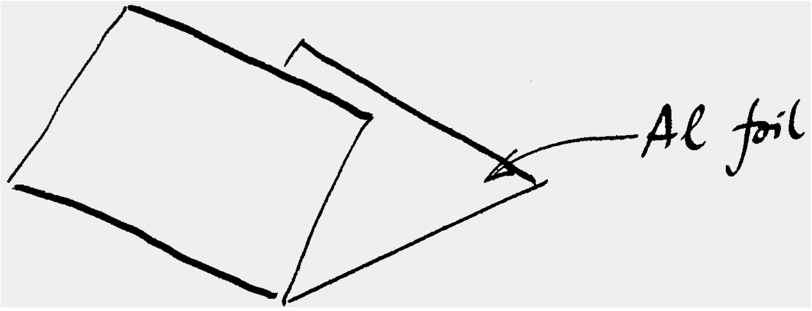
There are a number of safety issues associated with work at ISIS. The most obvious is the radiation hazard from the neutron beam and from irradiated samples and sample environment equipment. This is minimised by the use of interlocks, monitoring the radiation levels and appropriate handling and storage of irradiated materials. ***If in doubt, ask*** (your local contact, Health Physics or the Main Control Room). **Before you start an experiment you must watch the ISIS safety video** (in either the DAC or the coffee room in R3) and sign the yellow card. There is also the risk of exposure to chemicals, in this case the handling instructions on the back of the sample sheet state the required procedures to follow. Note that cadmium metal is toxic (it also activates in the beam) so should be handled with care. On removal from a cryostat, samples and centresticks are usually very cold, so should not be handled without gloves. Some of the sample environment equipment is heavy or bulky, so should be carried with due respect.

2.2 Preparing samples

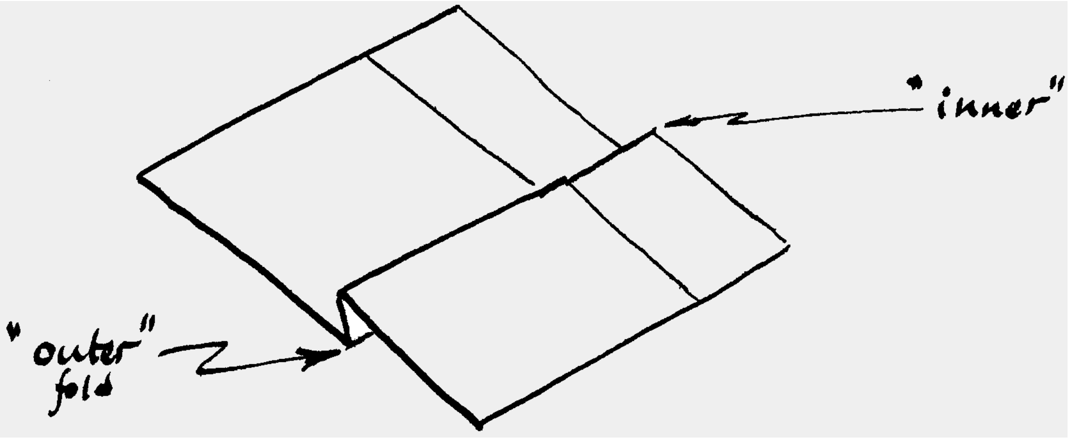
The laboratory's official handling instructions will be found on the back of the sample requirement form. You are required to observe them. For solids the easiest way to present the sample is to load it into an aluminium foil sachet. These are constructed as described in section 2.2.1. For liquids, a thin walled aluminium can is used. The same containers can be used for air or moisture sensitive samples, except that the can is loaded in a glovebox.

2.2.1 Preparing an aluminium sachet

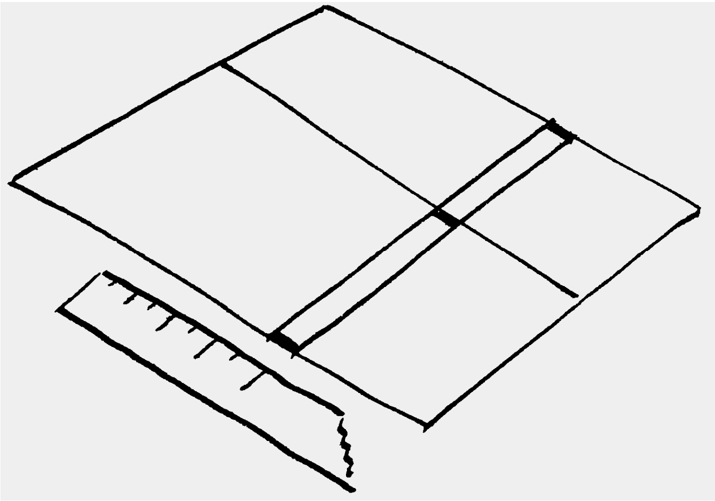
1. Tear off a piece of foil 20 cm long and fold it over



2. 1st ‘Z’ fold on right-hand side

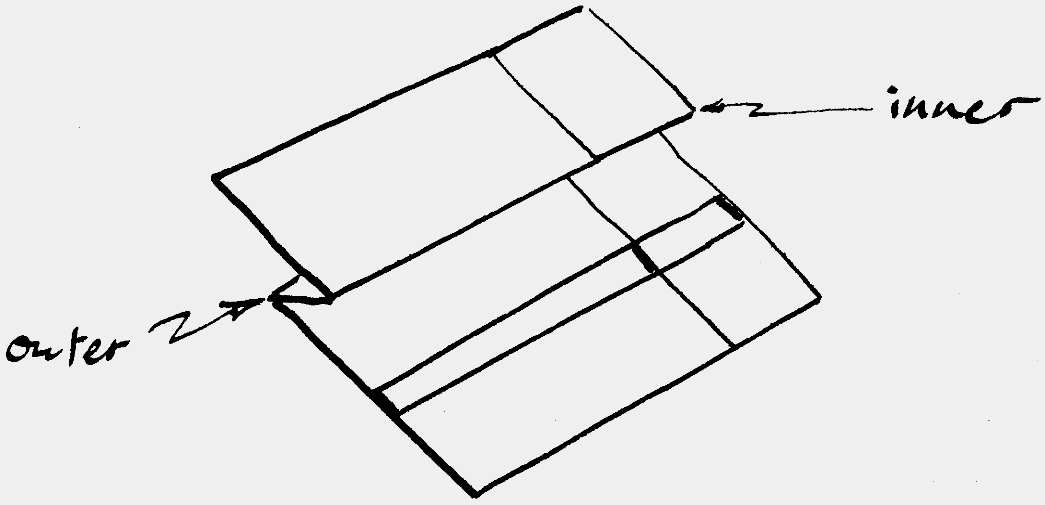


3. Press ‘Z’ fold flat, using back of fingernail, or plastic ruler.



4. From "outer" fold mark off sachet width (this is normally 40 mm; however, for bulky samples this must be ~50 mm).

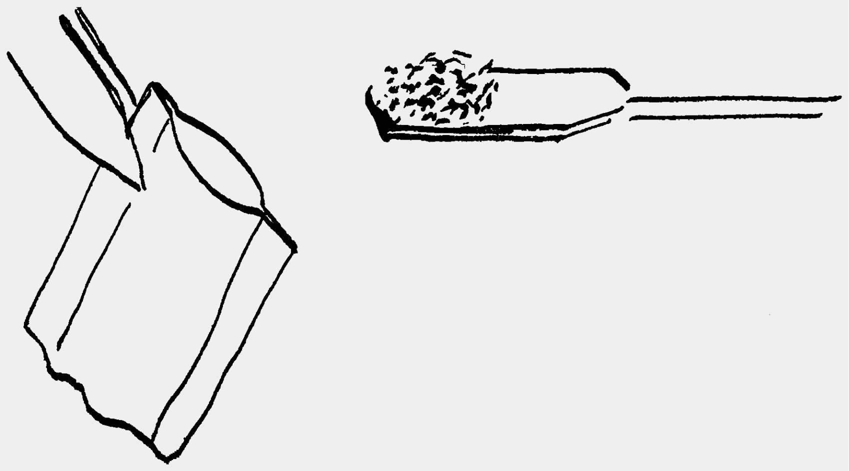
5. 2nd ‘Z’ fold, on left-hand side



6. Press Z fold flat and cut away excess foil from the sides.

7. Open the sachet with a pencil or spatula.

8. Fill the sachet. Note that it is often valuable to know exactly how much sample is in the beam, so weighing the sachet before and after filling it is good practice.



9. Tamp the sample gently to the bare of the sachet. Close off the sachet immediately above the sample using finger and thumb.

10. At a height of 40mm from the base of the sachet, fold a few times to seal.

11. The sample should be evenly distributed throughout the sachet using a cylindrical bottle like a "rolling pin".

Hints

1. Using a blunt pencil the sachet can be "impressed" with a name.

2. If you have produced a sample that is too thin, DO NOT start afresh, simply make

another and run both!

3. If you puncture a sachet, enclose this sachet in some Al foil which can be gripped on the centrestick as usual.

4. If you are using sachets on the samplechanger, ensure that they will fit within one of the aluminium frames

2.2.2 Preparing a liquid cell.

For liquid or air sensitive samples an indium wire-sealed thin-walled aluminium can should be used. With liquid samples the cell should be filled in a fume hood or a glovebox if sensitive to the atmosphere. For air or moisture sensitive solids, the sample should be loaded, and the can assembled, in the glovebox. There are two types available. One type of cell is designed to go on the 24-position samplechanger so up to 24 samples can be loaded simultaneously (these can be all cans or a mixture of sachets and cans). These cans use 1 mm indium wire. Figure 2.3 shows one of these cells disassembled and a complete cell. The cans have pathlengths of 1, 2, 3 or 5 mm, by suitable choice of the top-plate. For hydrogenous liquids, the 1 mm length should be used since this is sufficient sample to give an excellent spectrum in around 6 hours. The cells can be used either on the 24-position samplechanger or clamped to a standard centrestick.

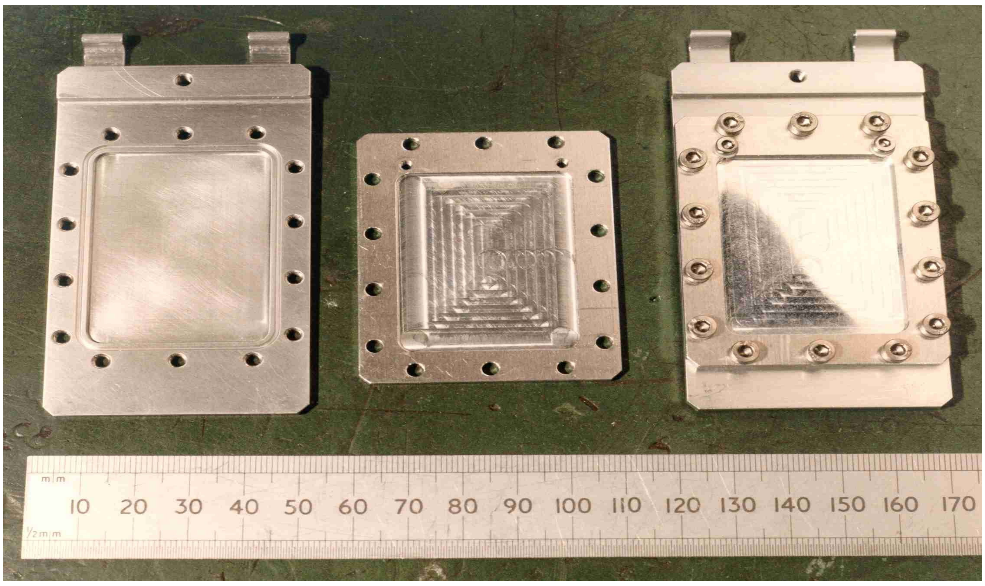


Figure 2.1: A liquid cell for the 24 position samplechanger. Left: baseplate, middle: top-plate, right: assembled.

Figure 2.2 shows the second ("HET") type cell of thin walled aluminium sample can and its components. The can consists of two outer cases and a spacer. The spacer thickness can be varied between 1 and 10 mm. The can is sealed using either indium wire (narrow grooves) or with Viton O-rings (wide grooves). Since the width of the can is much greater than that of the beam, solid samples should be loaded into a sachet and this positioned in the centre of the can before assembly. To reduce scattering from the cell, it should be completely shielded with cadmium apart from the opening at the front of the cell. Owing to the large mass of the cell, once loaded and attached to the centrestick, it should be immersed in liquid nitrogen for a few minutes immediately prior to putting the centrestick in the cryostat. This reduces the cool-down time to less than an hour, from several hours.

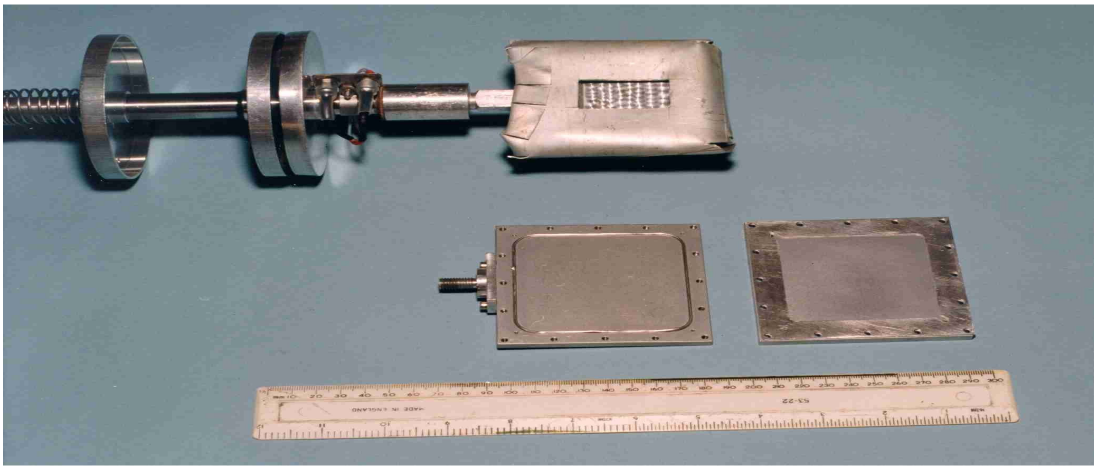


Figure 2.2: The components of a liquid cell and an assembled cell, shielded with cadmium mounted on a centrestick.

2.3 Loading the samples onto a centrestick.

For solid samples in sachets, the simplest method is to attach the sachet to the aluminium baseplate of an HET type can (lower left in Figure 2.2) with small strips of aluminium sticky tape. The beam centre is 1165 mm below the underside of the centrestick flange and the beam itself is 40 x 40 mm (h x w). The sample should cover as much of the beam area as possible and be preferably no more than 2 mm thick. If measurements at temperatures other than the base temperature of the CCR (~12K) are intended, then the best method is to attach heaters and a sensor to the sides of the plate. Note the sensor number! Care should be taken that only the sample and the Al sachet are in the beam; items such as sensors, heaters, tape or wire should not intrude. If it is intended to measure more than four samples at base temperature it is worth considering using the 24 position samplechanger. For six or more, its use is almost mandatory. Samplechanger type liquid cells are held by a clamp attached to the end of the centrestick (see *Figure 2.14*). HET cans have a mounting plate with an M8 screw that attaches to the end of the centrestick (top in *Figure 2.2*).

2.4 Loading the 24 position samplechanger

The 24 position samplechanger is shown in *Figure 2.3,* it consists of two parallel endless chains connected with horizontal bars from which the samples are suspended. The device can be controlled either manually from a handset attached to the control box in the electronics rack next to the services panel or under computer control. The latter is described in section 3.5. Note that samples can only be run at the base temperature of the cryostat (~20K with the samplechanger), it is not possible to put heaters on the sample or to heat the cryostat. You should also assume that the samplechanger can only be run forwards, so the samples should be loaded in the order in which you wish to run them.

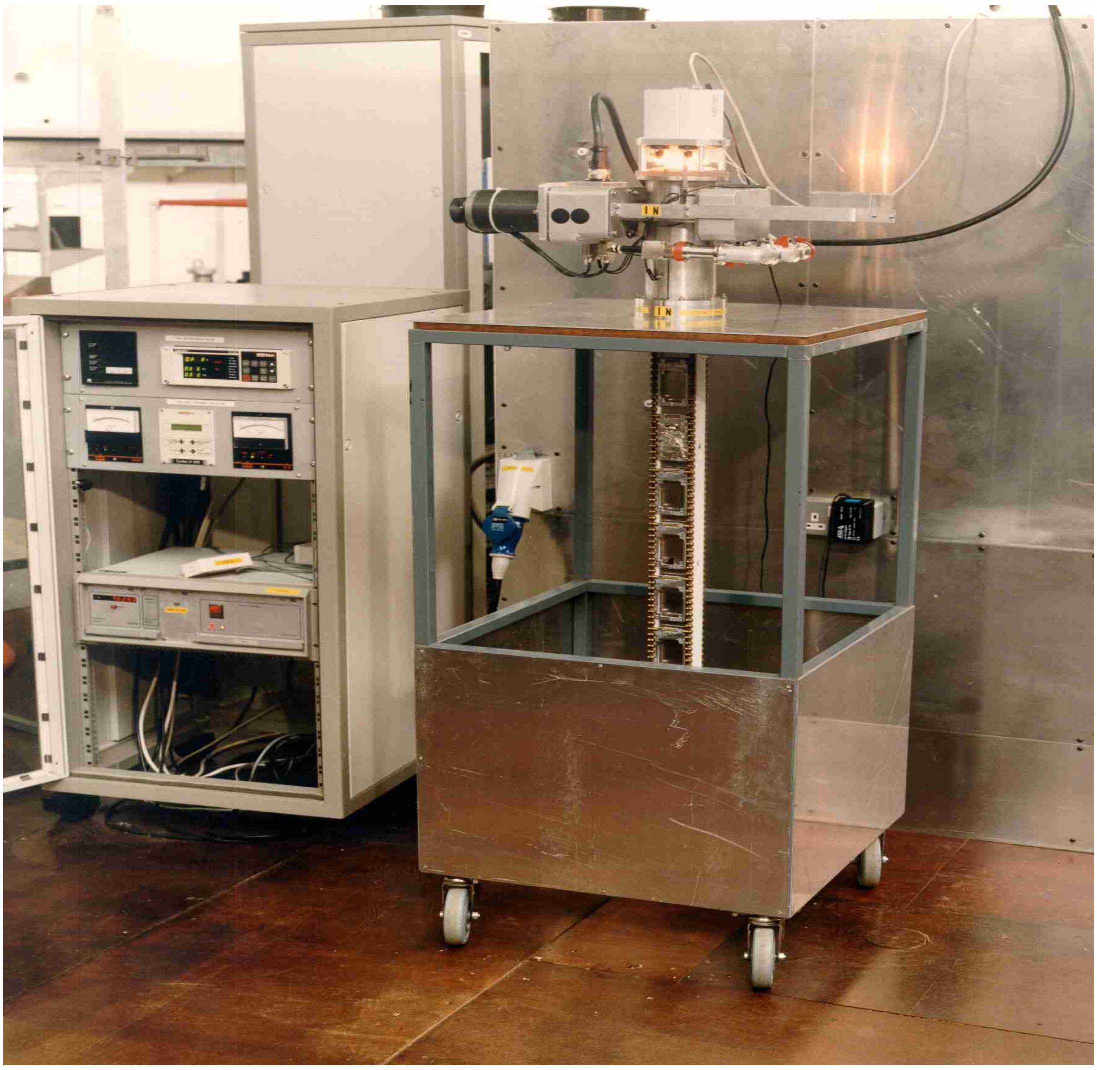


Figure 2.3: The 24 position samplechanger in its cradle (right) and its controller (bottom panel in electronics rack on the left).

The samplechanger can be loaded with sachets or liquid cells in any combination. Sachets are mounted on an aluminium frame and held on with aluminium sticky tape. The sachet must be held within the frame it should not protrude above it and the sticky tape should not be touch the chains. The right-hand side of Figure 2.4 shows a correctly mounted sachet To mount a liquid cell, it may be necessary to remove an aluminium frame. In either case, note the six digit number on the clamping plate.

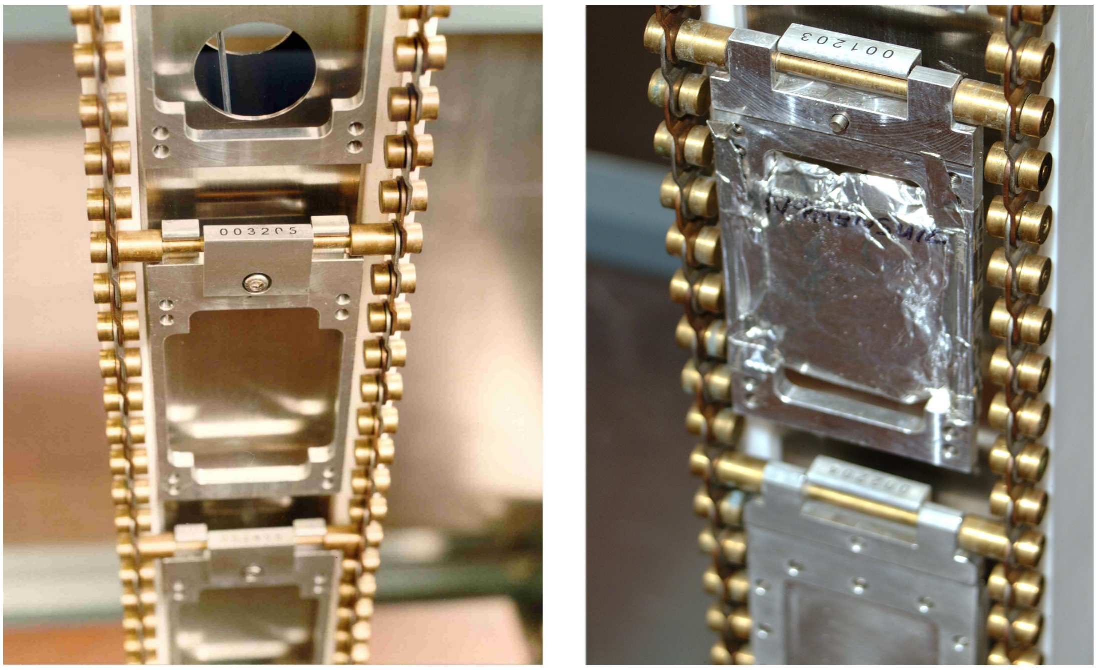

003205
001203

Figure 2.4: Loading samples on the 24 position samplechanger. Left: empty aluminium frame for a sachet, note the six digit number at the top of the clamping plate. Right: sachet mounted on a frame. A liquid cell is mounted at the position below it.

To move to a vacant position on the samplechanger, use the handset attached to the controller in the electronics rack, see Figure 2.5. There are four buttons on the handset: forward is the second from the top, back is the bottom button (the first and third buttons are not used), press and hold for either direction. There is a green status light to the right of the digital display on the control rack, the samplechanger will only move when this illuminated. When the sample is in the correct position, movement stops and the red "sample in lock" light is illuminated, there are also two red lights on the samplechanger that show the sample is in the correct position. The digital display will have changed by ~1600. To install or remove the samplechanger in TOSCA requires the use of the crane. **This must be done by the local contact.**

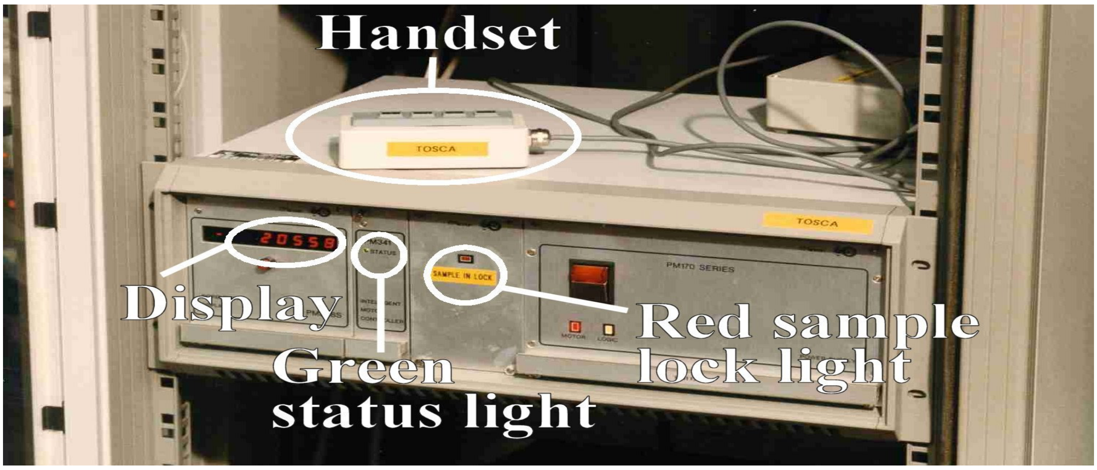

Handset
TOSCA
TOSCA
20558
STATUS
SAMPLE IN LOCK
PM170 SERIES
MOTOR
LOGIC
Display
Green status light
Red sample lock light

Figure 2.5: The 24 position samplechanger controller

2.5 Changing sample

Before changing a sample, familiarise yourself with the TOSCA cloche and the area around it. *Figure 2.6* shows the gate and interlocks to the TOSCA enclosure and highlights the location of the important items. The following procedure assumes that the four-position sample changer is being used. If an aluminium can or other large piece of equipment (e.g. a catalysis cell or a McWhan cell) is being used, then it is essential to pre-cool this in liquid nitrogen immediately before insertion into the cryostat, otherwise the cool-down time is prohibitively long. If the sample is pre-cooled in liquid nitrogen, then ensure that the lowest baffle does not come into contact with the liquid nitrogen because it can freeze in the cryostat and cause the centrestick to become stuck.

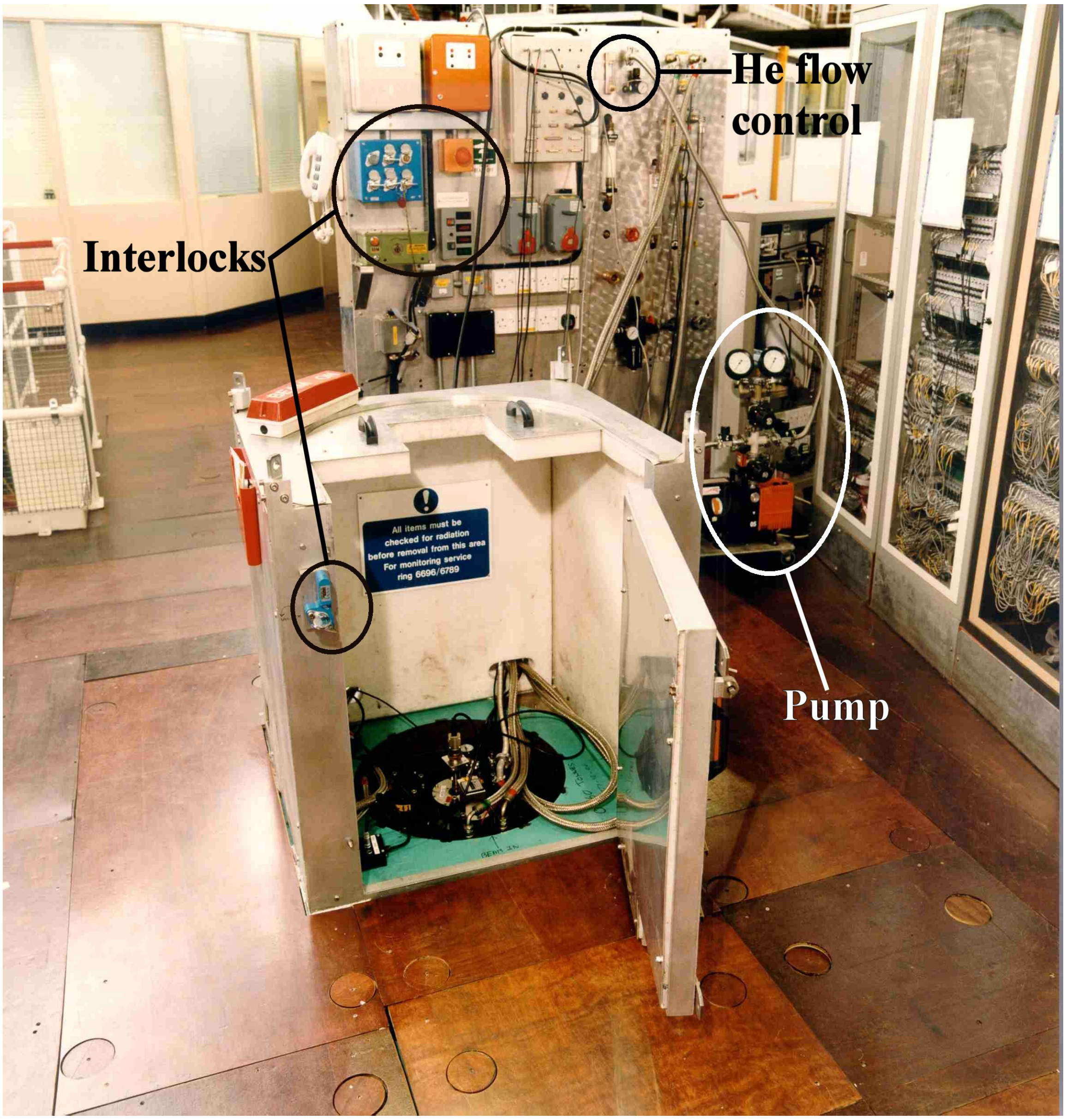

He flow control
Interlocks
All items must be checked for radiation before removal from this area For monitoring service ring 6696/6789
Pump

Figure 2.6: The TOSCA cloche and services panel showing the interlocks interlocks, pump and He flow control.

Access to the TOSCA CCR is via the "cloche" located on the mezzanine floor. This is interlocked by the standard ISIS key system. The interlocks on the instruments are there to try to make it impossible to get close to the neutron beam. There are two sets of interlock keys: The Master (‘M’) key, which is to be found on the front of the Green box and is labelled with a red tag; The remainder are ‘S’ keys which are located in the Blue box, see *Figure 2.7*. There are two shutter control boxes: one in the cabin and one on the services panel behind the cloche.

Note: You only have control of the shutter if the Master key is in the Green box. If you try to operate the shutter whilst the interlocks are not complete you will trip-off ISIS.

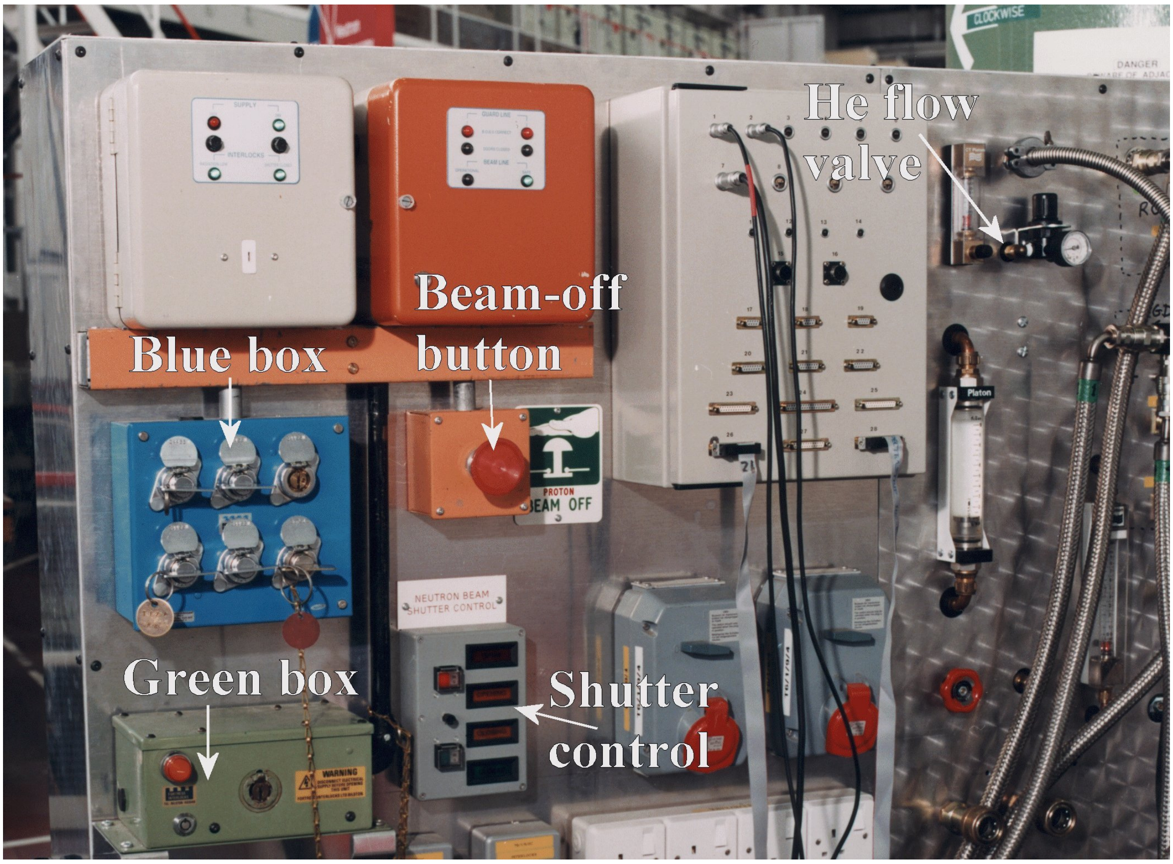

He flow valve
Beam-off button
Blue box
Green box
Shutter control
CLOCKWISE
DANGER
INTERLOCKS
PROTON BEAM OFF
NEUTRON BEAM SHUTTER CONTROL
WARNING
Platon

Figure 2.6: TOSCA services panel showing the Green (bottom left) and Blue (centre left) interlock boxes and the shutter control (bottom middle).

The Master key is only released when the neutron shutter is closed. Conversely the shutter can only be opened if the key is in place in the Green box.

The ‘S’ keys give access to the sample enclosure and other controlled areas. They can be released by placing the Master key in the bottom right hand slot of the Blue box. This is normally the only vacant slot.

1. "END" the current run (the Dashboard changes to "SETUP") and write: the time, Run No and total number of microamps used into the Instrument Diary.
2. Close the shutter to the beam (green button marked "CLOSE" on the Neutron Beam Shutter Control Panel located on the services panel by TOSCA, *Figure 2.7*, and in the cabin *Figure 2.8*. The shutter takes a few minutes to close.

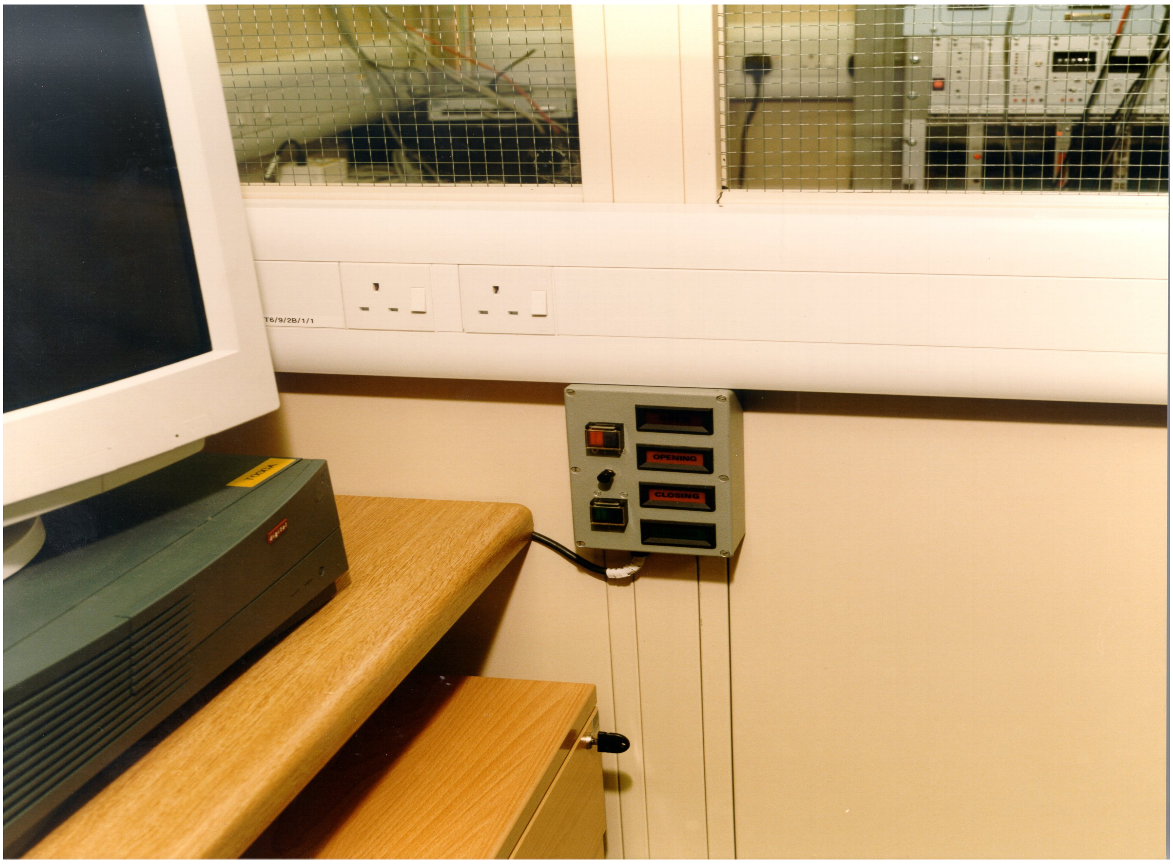


Figure 2.8: The shutter controls in the TOSCA cabin.

3. Ensure the shutter is **closed.** Wait until both the blue fluorescent light, and the red "Beam On" sign are off and the green "CLOSED" light is illuminated in the shutter control panel. The radiation monitor on the wall of the target station must show a green light and a reading of less than 20 µ Sv/hr, *Figure 2.9.*

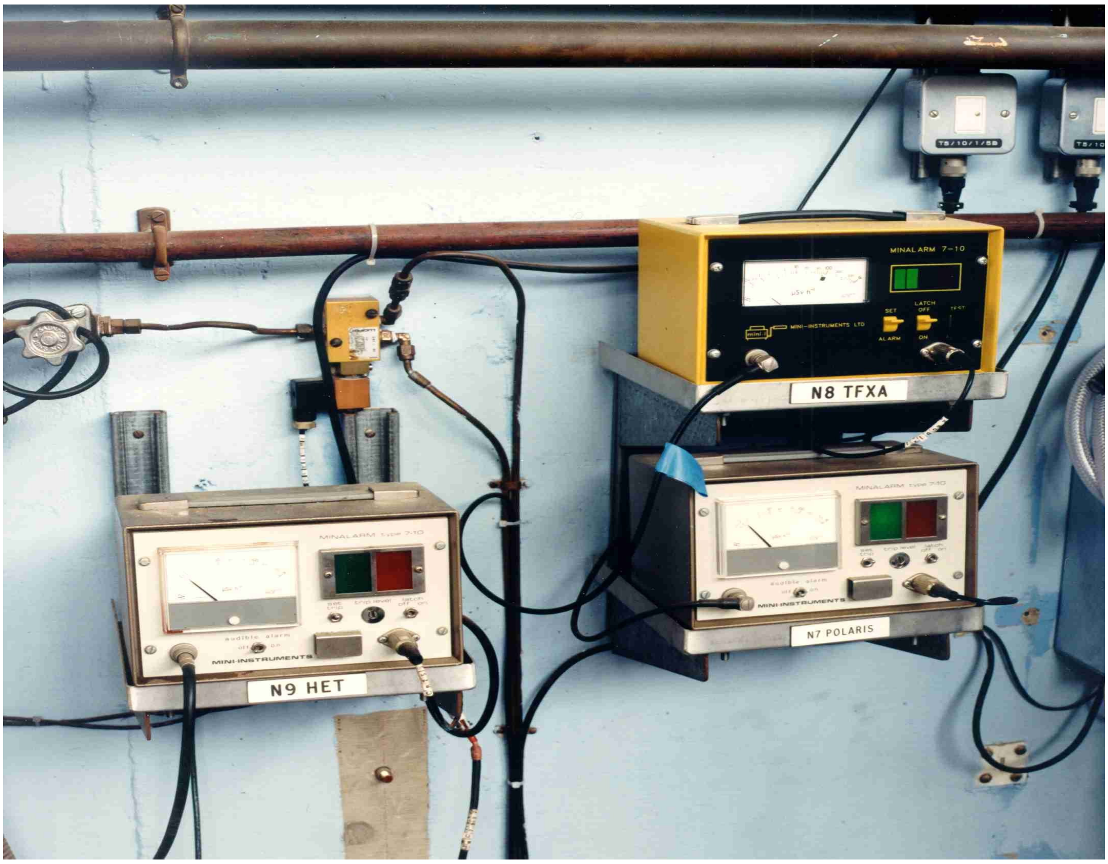


Figure 2.9: The radiation monitor on the target station wall. TOSCA's is labelled "N8 TFXA" (upper right).

3. **Turn** anti-clockwise the "Red" (i.e. carrying a red tag) key in the "Green" box and release it.
4. Engage the Red key in the "Blue" box, and turn it clockwise.
5. This liberates all other keys in the Blue box. Select any other key and turn it anti-clockwise and remove it.
6. Remove the shielding from the top of the cloche.
7. Place the key in the enclosure lock and turn it anti-clockwise.
8. Rotate the bolt fully and withdraw. the bolt fully and withdraw.
9. Open fully the small brass valve on the flowmeter to the helium cylinder (see Figure 2.6). Do not adjust the regulator, the gas pressure on the gauge should read about 0.5 bar.
10. Open valve No's 1, 2 and 4 on the cryostat pump (see *Figure 2.10*) The vacuum gauges should show the pressure rising slowly, meanwhile the He gas flow is at maximum. Ensure that the light blue valve on the CCR is pointing vertically, *Figure 2.13*.

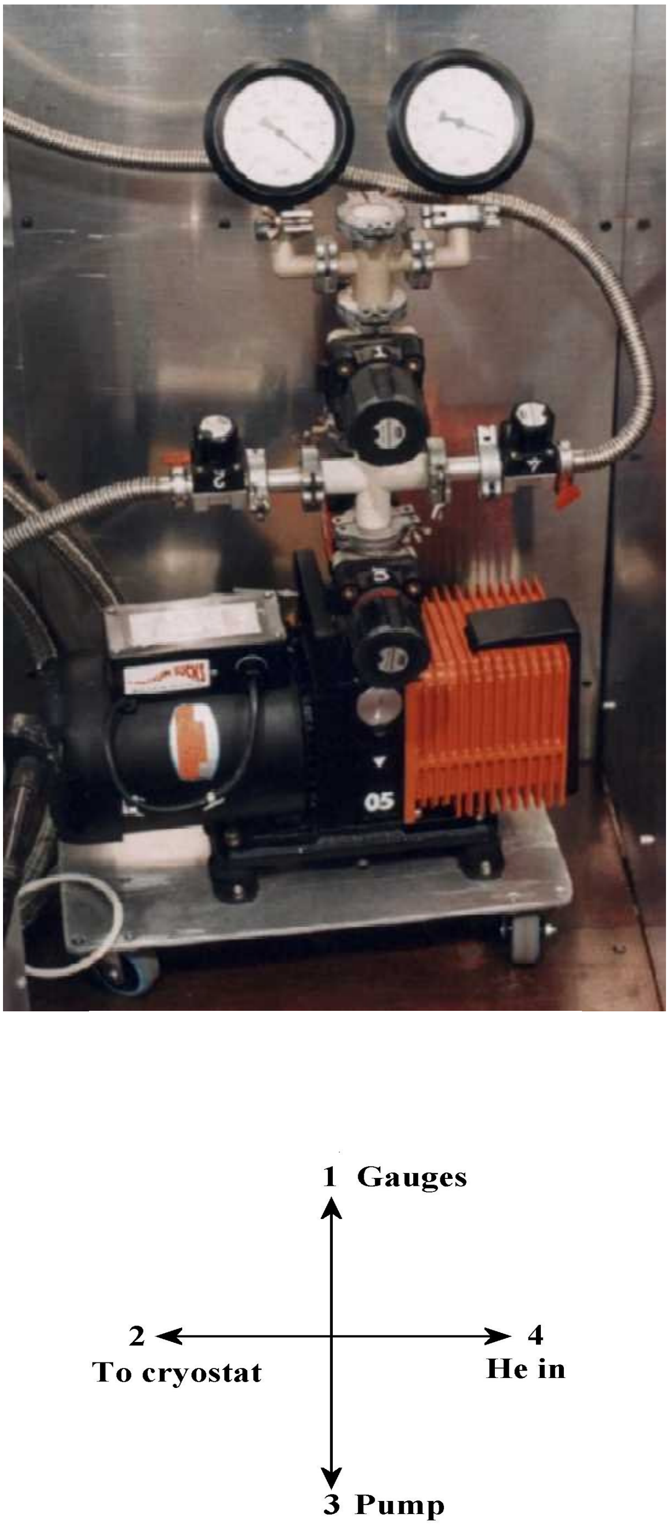

1 Gauges
2 To cryostat
4 He in
3 Pump

Figure 2.10: The pump manifold and sketch showing the numbering of the valves.

11. Close valve 2 when the vacuum gauge shows 1000 mbar.

Note: *Because the vacuum gauge on the cryostat pump will automatically release excess pressure: VALVE 4 MUST BE CLOSED.*

12. Remove the thermometer connection by unscrewing the upper "silver" knurled nut, *Figure 2.11*. DO NOT UNSCREW THE LOWER BRASS NUT.

13. Unscrew the retaining bolts at the top of the sample centrestick and withdraw the centrestick smoothly but RAPIDLY. *(Figure 2.12*).

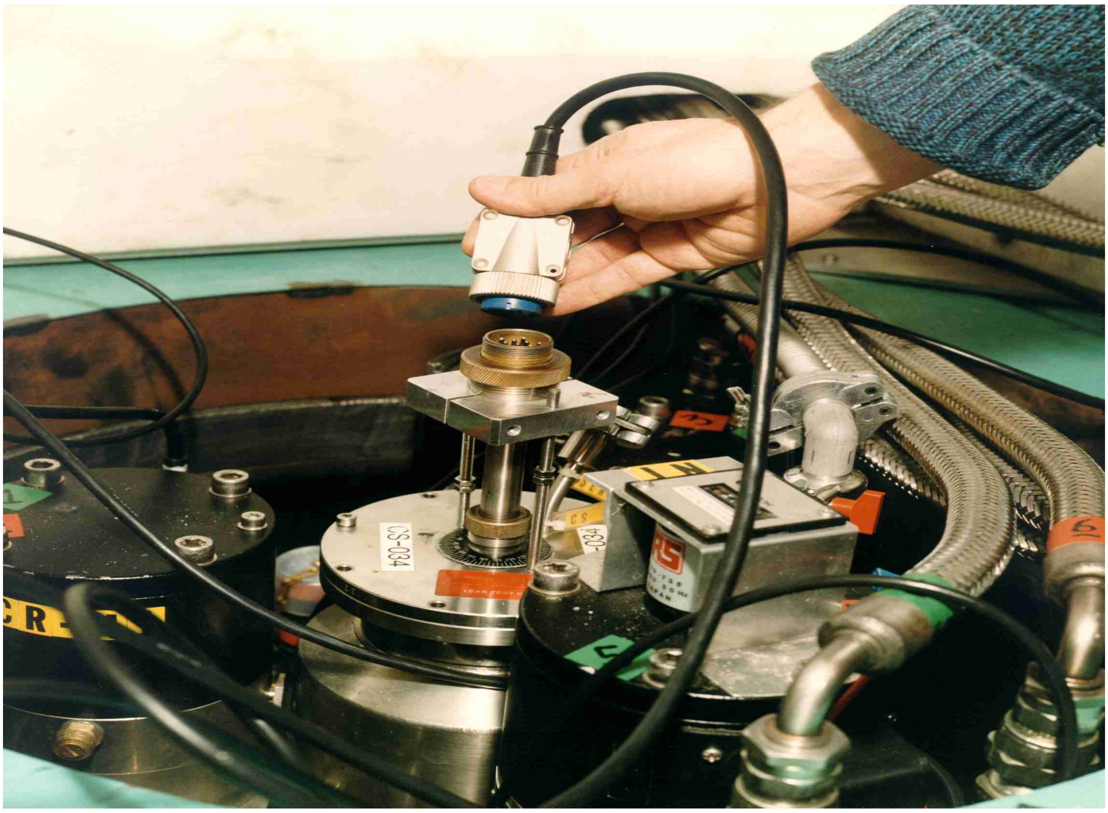


Figure 2.11: The thermometer connection on the CCR.

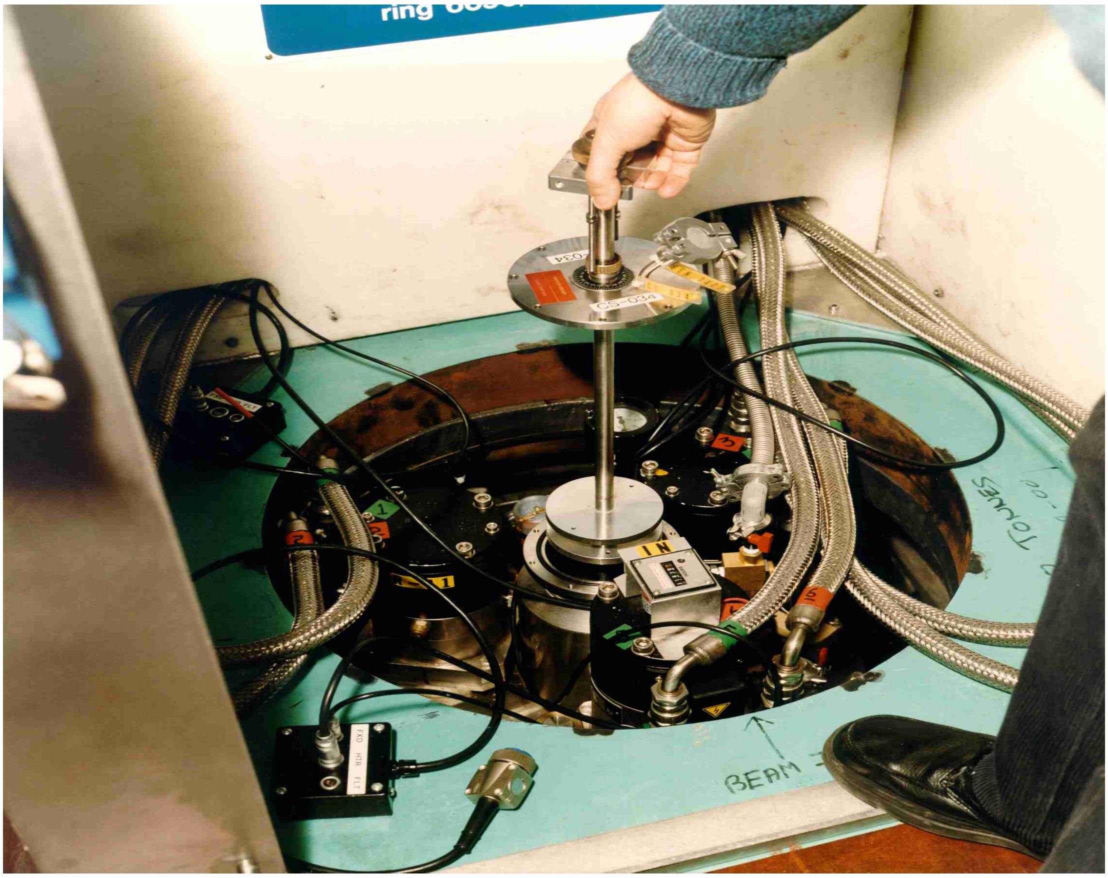


Figure 2.12: Illustrating centrestick withdrawal

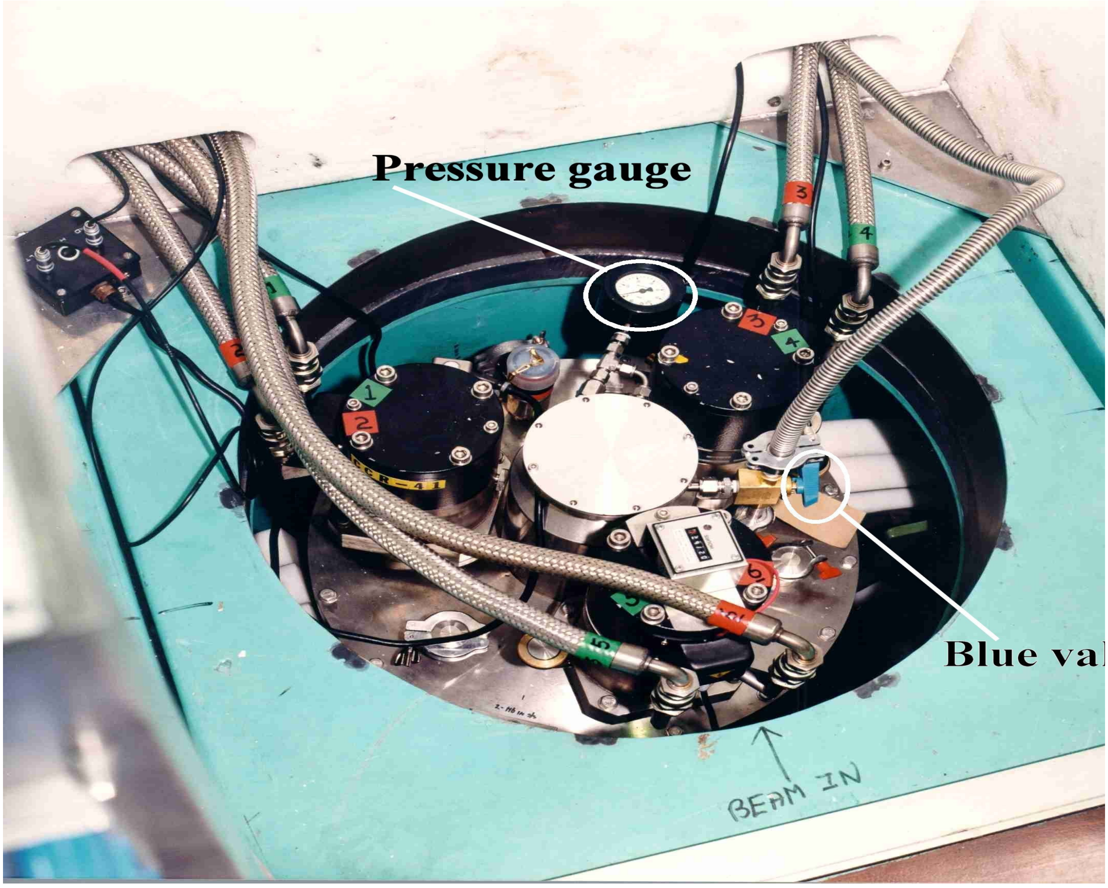


Figure 2.13: Photo graph of the cryostat with the centrestick withdrawn and blanking flange in place. The blue valve on the side of the CCR and the pressure gauge are labelled.

14. Cover the cryostat top with the blanking plate and bolt it down *(Figure 2.13*). If it will be more than a few minutes before the next sample is inserted, the helium should be pumped out, see 19 and 20. This keeps the cryostat cold and reduces cool-down time for the next sample.
15. Take the centrestick to the work bench and replace the old sample with new sample (see Section 2.2 and 2.6). If a different centrestick is to be used, ensure that the sample is inside the lead castle on the work bench.
16. Unscrew bolts retaining blanking plate, the pressure falls to 0.
17. Remove the blanking plate and push the centrestick down into the cryostat, RAPIDITY is needed but CARE must be used. Bent centresticks are expensive to replace.
18. Secure centrestick lid with bolts.
19. Switch on the cryostat pump (switch on right hand side of pump).
20. Open valves 2 and 3, vacuum gauge begins to register.
21. WAIT until the pressure drops to 25 millibar on the gauge on top of the CCR.
22. Close the blue valve on the CCR and valves 2 and 3 and switch off the pump.

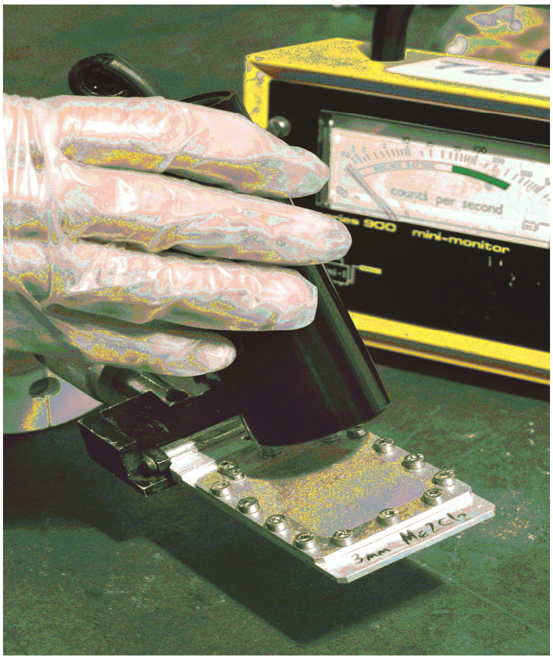
23. Reconnect the thermometer cable to centrestick.
24. Check which sample is oriented correctly (usually perpendicularly) with respect to the neutron beam.
25. Close the interlocked door (do steps 4 - 8 in reverse.)
26. Open the shutter and start collecting data (see section 4: Controlling the instrument).

2.6 Removal from centrestick

This work should be done with the sample centrestick being on the TOSCA centrestick stand, on the work bench on the mezzanine floor.

27. Turn the hot air blower on and warm the sample.
28. Release the sample sachet from the cadmium lantern using long-nose pliers to remove the retaining wire or aluminium tape. Remember that cadmium metal strongly activates in the neutron beam.
29. Remove the sample using tweezers - or - if you must, gloved hands.

NEVER HANDLE ACTIVE SAMPLES WITH BARE HANDS.



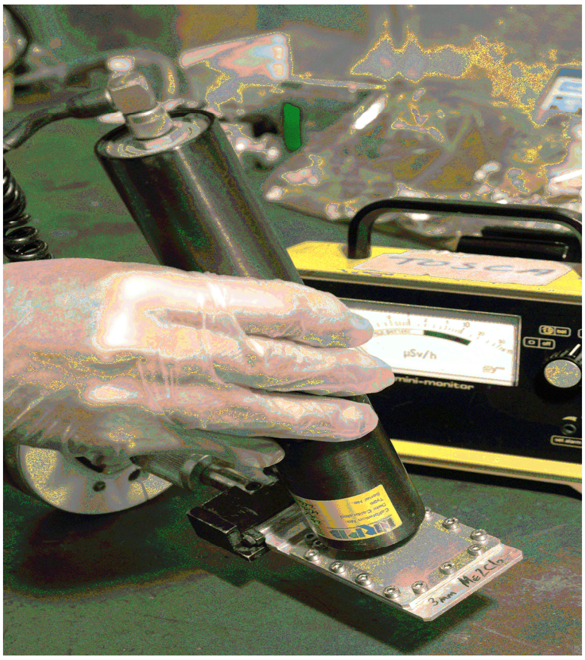


Figure 2.14: Testing the sample with β (top) and γ (bottom) radiation monitors.

4. MONITOR the sample with both β and γ monitors (β monitor cap off, see *Figure 2.14*). If the radiation level is less than 75 µSv consign the sample to the TOSCA active sample cupboard. If the levels are greater than 75 µSv inform the **Duty Officer** for instructions (ext: 6789)

Samples consigned to the active cupboard **MUST** be in sealed plastic bags and labelled with the owner's and sample names and date. The sample environment form should also be included. Spare bags are in the tool cupboard and the prep. labs.

Note: *If you really must transfer active loose powders between sample holders or if a sachet bursts accidentally, phone the Duty Officer for instructions and help.*

-

NO SAMPLES MAY BE REMOVED FROM ISIS WITHOUT THE CONSENT OF HEALTH PHYSICS.

2.7 Removal of a Stuck Centrestick

Occasionally, during removal from the CCR a centrestick is found to be stuck in the cryostat. There are a number of possible causes of this, of which the most common are failure to ensure that the centrestick is dry when it goes into the cryostat, a leak around the top flange of the centrestick caused by the flange being incorrectly seated on the O-ring, or if the sample was pre-cooled in liquid nitrogen, solid nitrogen gluing the lowest baffle to the cryostat wall. By whatever means, the usual result is a small amount of air or nitrogen condensing between the baffles of the centrestick and the cryostat wall. In these cases, warming the cryostat to 90K is sufficient to free the centrestick. If ice is present, then it is necessary to warm it to near room temperature.

THE FIRST ACTION SHOULD BE TO INFORM YOUR LOCAL CONTACT.

The sequence of actions is:

5. Fill the centrestick chamber with helium gas. ***The flange of the centrestick must be bolted down.***
6. Switch off the two CCR compressors labelled TOSCA on the ground floor by the outer wall of R55 *inside* the hall, *Figure 2.15*. **DO NOT TOUCH** the five compressors that are outside the hall, *Figure 5.2*.
7. Warm the cryostat to 90K.
8. When the sample temperature is 90K attempt to remove the centrestick as normal (see section 2.5)
9. If the centrestick cannot be removed, wait until the cryostat has reached room temperature.
10. When the centrestick has been removed, replace the blanking flange and flush the sample volume with helium gas three times before installing the next sample.
11. Re-start the CCR compressors.

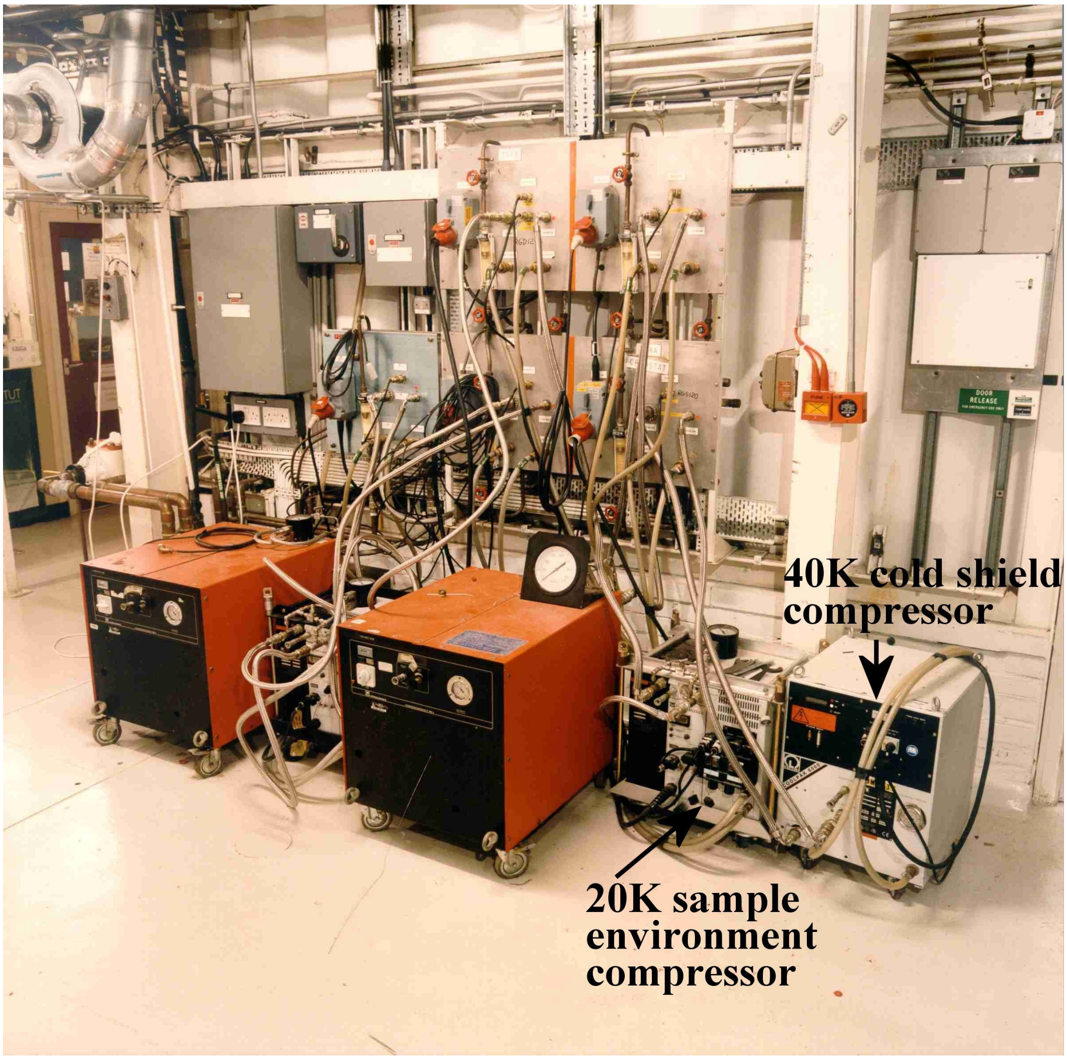

40K cold shield compressor
20K sample environment compressor

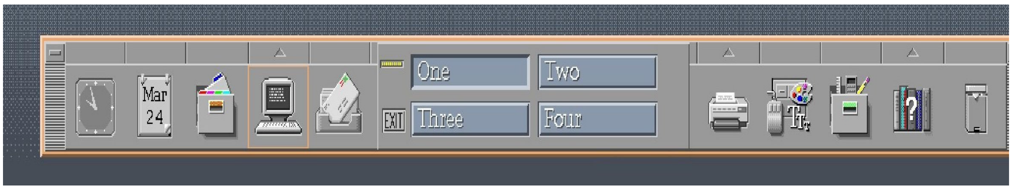
Figure 2.15: TOSCA CCR compressors inside the hall. Right: 40K cold shield compressor, left: 20K sample compressor.

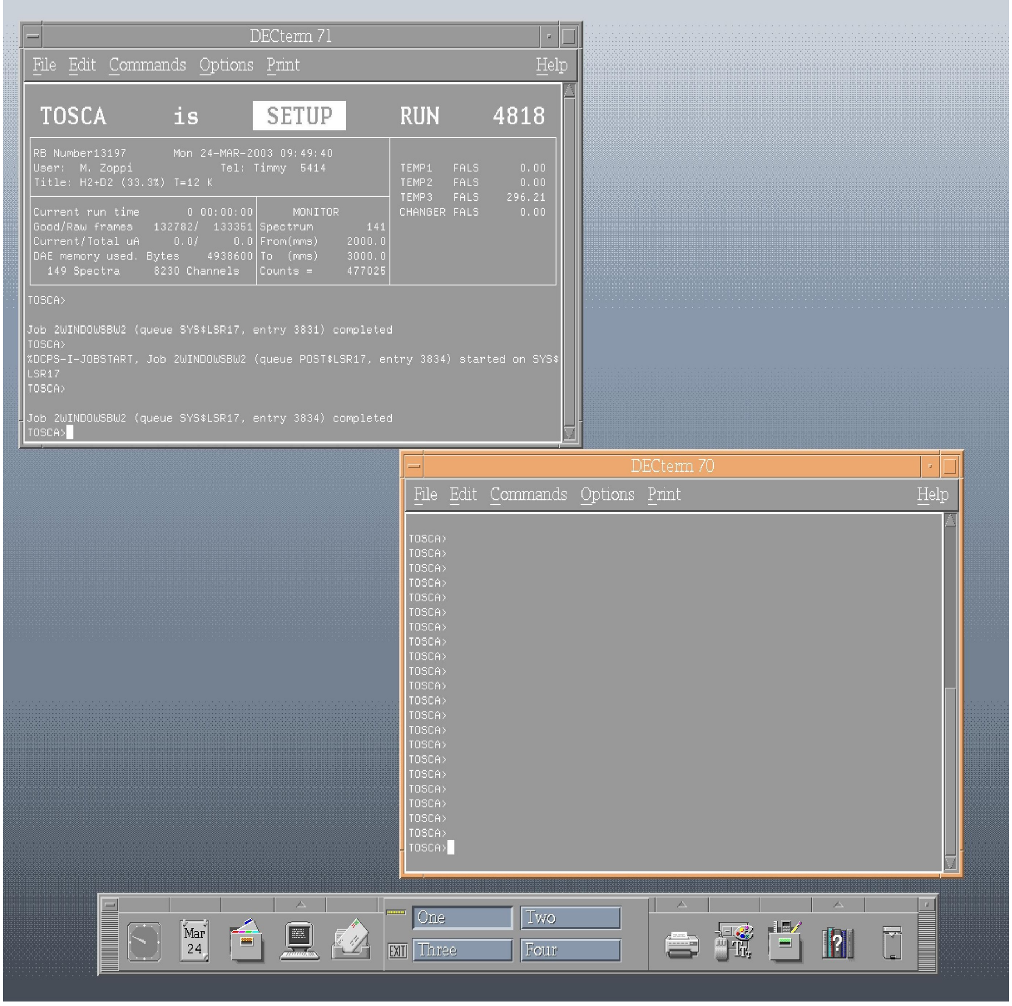
3. CONTROLLING THE INSTRUMENT

TOSCA is run by a DEC-ALPHA 500 computer located in the TOSCA cabin on the mezzanine level of R55. After logging-on (if necessary) the screen will look like *Figure 3.1*. In the centre of the toolbar at the bottom of the screen are four buttons labelled one to four. Each of these has its "own" screen associated with it and each screen can have as many windows as desired. The convention that is used is:

Screen 1 is used for displaying the instrument status (the "Dashboard") and controlling the instrument
Screen 2 is used to display data using GENIE
Screen 3 is for OPENGENIE
Screen 4 is for the users e.g. to telnet to their home computer.

To create a window, on the toolbar click on the icon for a terminal (4th from the left in *Figure 3.1*), a menu then pops-up and click on the item labelled DECTERM. This procedure is the same in any of the screens.


Mar
24
One
Two
EXIT
Three
Four


DECterm 71
File Edit Commands Options Print Help
DECterm 70
File Edit Commands Options Print Help
Mar
24
One
Two
EXIT
Three
Four

```
TOSCA      is      SETUP      RUN      4818

RB Number13197       Mon 24-MAR-2003 09:49:40
User:  M. Zoppi             Tel: Timmy  5414      TEMP1   FALS      0.00
Title: H2+D2 (33.3%) T=12 K                       TEMP2   FALS      0.00
                                                  TEMP3   FALS    296.21
Current run time       0 00:00:00|    MONITOR     CHANGER FALS      0.00
Good/Raw frames   132782/  133351|Spectrum      141
Current/Total uA      0.0/      0.0|From(mms)    2000.0
DAE memory used. Bytes    4938600|To  (mms)    3000.0
  149 Spectra     8230 Channels  |Counts =     477025
TOSCA>

Job 2WINDOWSBW2 (queue SYS$LSR17, entry 3831) completed
TOSCA>
%DCPS-I-JOBSTART, Job 2WINDOWSBW2 (queue POST$LSR17, entry 3834) started on SYS$
LSR17
TOSCA>

Job 2WINDOWSBW2 (queue SYS$LSR17, entry 3834) completed
TOSCA>
```

```
TOSCA>
TOSCA>
TOSCA>
TOSCA>
TOSCA>
TOSCA>
TOSCA>
TOSCA>
TOSCA>
TOSCA>
TOSCA>
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TOSCA>
TOSCA>
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TOSCA>
TOSCA>
TOSCA>
TOSCA>
TOSCA>
TOSCA>
```
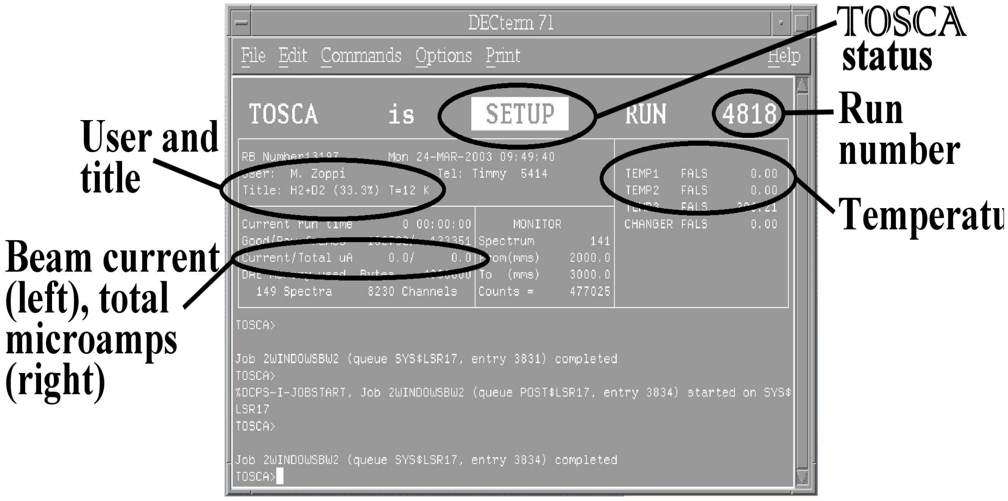

Figure 3.1:Top: the toolbar on the TOSCA terminal in the cabin, bottom: Screen 1 showing the Dashboard and the Control window.


TOSCA status
Run number
Temperatu
User and title
Beam current (left), total microamps (right)
DECterm 71
File Edit Commands Options Print Help

```
TOSCA      is    SETUP      RUN     4818

RB Number13197        Mon 24-MAR-2003 09:49:40
User:  M. Zoppi            Tel: Timmy  5414       TEMP1   FALS      0.00
Title: H2+D2 (33.3%) T=12 K                       TEMP2   FALS      0.00
                                                  TEMP3   FALS    [illegible]
Current run time          0 00:00:00   MONITOR    CHANGER FALS      0.00
Good/Raw frames  [illegible]/  433351 Spectrum         141
Current/Total uA    0.0/       0.0 From(mms)    2000.0
DAE memory used  Bytes  [illegible] To  (mms)    3000.0
  149 Spectra     8230 Channels   Counts =     477025

TOSCA>

Job 2WINDOWSBW2 (queue SYS$LSR17, entry 3831) completed
TOSCA>
%DCPS-I-JOBSTART, Job 2WINDOWSBW2 (queue POST$LSR17, entry 3834) started on SYS$
LSR17
TOSCA>

Job 2WINDOWSBW2 (queue SYS$LSR17, entry 3834) completed
TOSCA>
```

Figure 3.2: The Dashboard on the TOSCA terminal in the cabin.

For screen 1 two DECTERMs are needed. At the `TOSCA>` prompt type:

```
TOSCA>stat on ←
```

(← means "press enter") and the Dashboard will appear, *Figure 3.2*. The useful data in the screen are:

- The run number at the top right.
- The instrument status in the centre, this can be SETUP for changing samples, RUNNING for collecting data or WAITING for a control parameter (usually the temperature) to be true.
- The user name(s) and sample title are at the left, halfway down.
- Below this is the ISIS beam current to the target and on the same line is the total microamps ("Total uA") received in the current run. For adequate statistics from a hydrogenous sample this should be at least 600 (3 - 4 hours runtime), small samples or non-hydrogenous ones will require considerably longer.
- On the right is the sample temperature ("TEMP") in Kelvin, below this is the cryostat temperature ("TEMP1" in K).

The second window is the TOSCA control window, which should only be used for control commands such as beginning, updating and ending runs, changing temperature and starting instrument control command files. This terminal should be left in the `tosca$disk0:[tosca]` area at all times.

Note: *Do not leave files in the* `tosca$disk0:[tosca]` *area that you want to keep. The area is regularly purged.*

3.1 Change

The change command allows the user to edit the Dashboard information. Typing the command

`TOSCA> change` ← (can be abbreviated to `cha`)

will initiate the Dashboard editor. Move between areas using the up and down cursor keys and over type. There are six pages, you will only modify the first. This page contains title and user information. When entering the title please be informative; abbreviations or sample numbers are not helpful. The accumulated spectra on TOSCA form a unique library whose usefulness is compromised if the spectra are not clearly identified.

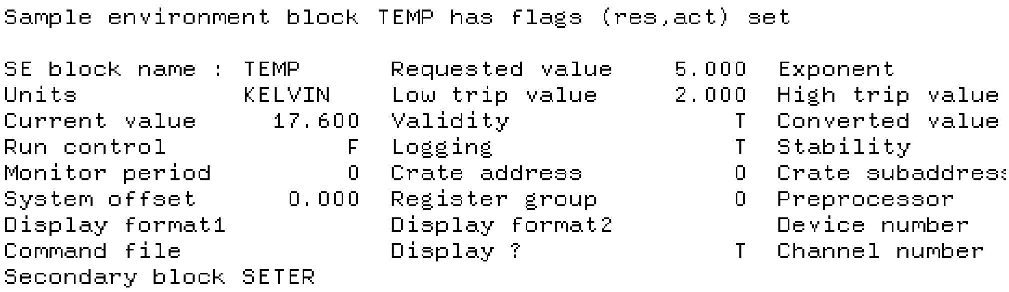
To exit press `[PF1]` (found on the numeric keypad on the right of the keyboard). A prompt will appear at the top of the screen, to exit press `[e]`.

3.2 Setting sample environment parameters

The top right hand portion of the Dashboard displays the sample environment parameters sample temperature (normally TEMP) and cryostat temperature (normally TEMP1). If you have changed centresticks or are using a sensor attached to the sample rather than the one built into the centrestick, you will need to input the sensor number. Each sensor is individually calibrated and a unique four digit number is written on the sensor. On each centrestick is a label with "SEN" on it that gives the sensor number. To check the censor number type:

`TOSCA> cshow temp/full` (or `temp1`)

The computer will respond:



```
Sample environment block TEMP has flags (res,act) set

SE block name :  TEMP       Requested value     5.000  Exponent
Units            KELVIN     Low trip value      2.000  High trip value
Current value      17.600   Validity                T  Converted value
Run control             F   Logging                 T  Stability
Monitor period          0   Crate address           0  Crate subaddres
System offset       0.000   Register group          0  Preprocessor
Display format1             Display format2            Device number
Command file                Display ?               T  Channel number
Secondary block SETER
```

The "Device number" is the sensor number. To input a different sensor number type:

```
TOSCA> cset temp/devspc=xxxx
```

Where xxxx is the sensor number.

Most spectra are recorded at the base temperature of the cryostat. If other temperatures are required, then cartridge heaters need to be attached to the sample before it is loaded into the cryostat. To change the sample temperature the cset command is used as follows:

```
TOSCA> cset temp/value=1OO/control
```

will set the sample temperature to 100K

Limits can be set to ensure that data are only collected between specified temperatures:

```
TOSCA> cset
temp/value=45/lolimit=40/hilimit=50/control
```

will ensure that data is only collected while the sample temperature lies between 40K and 50K. If the sample temperature strays out of these limits the instrument will be put into "WAITING" mode.

If run control is no longer required the nocontrol qualifier should be used:

```
TOSCA> cset temp/nocontrol
```

When measurements are to be made at base temperature, the heater is usually switched off. If you want to warm up the sample, and there appears to be no response to the cset temp command, check that the heater is plugged in and switched on. In the TOSCA cloche, the heavy black cable must be plugged into the socket marked "HTR" on the same black box to which the temperature sensor lead is attached. The heater on/off switch is on the Eurotherm crate, in the electronics rack in the TOSCA cabin. The % of the maximum power is also controllable. To determine the current setting type:

```
FEM> cshow max_power/enq
```

The computer will return:

```
Value returned was xx
```

Where xx is the % power. To change this value type:

```
FEM> cset max_power ??
```

Where ?? is the desired value (0 ≤ ?? ≤ 100)

3.3 Data collection commands

All the following instrument control commands may be abbreviated to three letters.

`begin`	Starts a run.
`update`	Stores the data collected so far in the current run parame (CRPT)
`store`	Stores the data collected up to the last update in the file `tosca$disk0:[tfxmgr.data]TSCA0<xxxx>.sav`. T command should always be preceded by an update
`pause`	Pauses data collection.
`resume`	Resumes data collection.
`abort`	Aborts the current run without saving any data.
`end`	Ends the current run and stores the data in `TOSCA$disk0:[tfxmgr.data]TSC0<xxxx>.raw` The analysed automatically by a batch program when a run is process takes a few minutes, after that it can be viewed u

3.4 Using command files

Command files are written to control the instrument. An example is:

`$ begin`	begins run
`$ waitfor 1000 uamps`	waitfor1000
`$ end`	end run
`$ cset temp/value=80/lolimit=75/hilimit=85/control`	sets temper
`$ wait 00:40:00`	wait 40 min (temperatur stabilisation
`$ change title """Sample at 80K"""`	title change <u>essential</u>)
`$ begin`	begins run
`$ waitfor 1000 uamps`	spaces are
`$ end`	
`$ exit`	

Command files are created using the VMS editor and end with the extension .com.

They are run from the TOSCA Control window using @< filename>. To interrupt a command file type [Control] Y. Note that you are unable to use the window when a com file is running.

Note: The two commands WAIT and WAITFOR are different, and confusion over their use is one of the main causes of command file failures.

```
WAIT
```

This is a VMS command that waits for a specified time. The time must be given in the hrs:mm:secs format used by the VMS operating system e.g.

```
WAIT 01:30:00
```

will wait for one hour 30 minutes before executing the next command.

```
WAITFOR
```

This is an instrument control command and can be used to wait for certain amount of data to be collected. The most common usage of the command is to wait for a certain number of microamps, in this case the suffix uamps must be given after the number e.g..

```
waitfor 1000 uamps
```

will wait for 1000 microamps of beam current before executing the next command. If ISIS goes off then it will sit and wait! Note that the format is rigid, there must be a space between the number and "uamps".

A common requirement is to wait until the sample reaches a given temperature. This can usually be estimated fairly accurately and a command file with a `WAIT` statement used. Alternatively, the `cset` command with the `/control` option can be used, but data is not collected while in the WAITING state. Instead, if the `/chklog` option is used with `cset` then the next command is only executed when the condition is true.

A sample command file is:

`$ cset temp/value=20/lolimit=5/hilimit=25` *sets temperature limits*
`$ begin`
`$ cset temp/chklog`
waits until the temperature is in the range 5 - 25K
`$ end`

end run

```
$ change title """Sample at 20K"""
$ begin
```

begins new run

```
$ exit
```

This will collect data until the temperature is in the range 5 - 25K, when it ends the run, changes title and starts a new run. The commonest use for this command is while waiting for the sample to cool, but it is not the only possible use.

Command files are created using the VMS editor and end with the extension `.com`. They are run from the TOSCA Control window using:

```
@ <filename>
```

To interrupt a command file type [Control] Y. Note that you are unable to use the window when a com file is running.

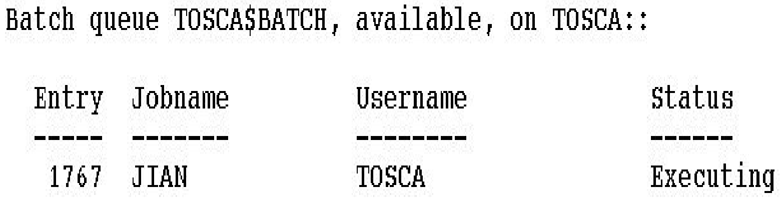
A better way to run a command file is to submit it to`TOSCA$BATCH` by:

```
submit/que=tosca$batch ←
```

The advantages are that your local contact can interrupt it and modify it remotely if necessary, this is not possible if `@ <filename>` is used. Also, in theory at least, if the TOSCA computer crashes, the batch job should restart, using `@ <filename>`, the command file is terminated. To check on the status of the command file use:

```
sho que tosca$batch ←
```

The response will be:



```
Batch queue TOSCA$BATCH, available, on TOSCA::

  Entry  Jobname         Username            Status
  -----  -------         --------            ------
   1767  JIAN            TOSCA               Executing
```

where `Jobname` is the name of the command file. To stop a batch job type:

```
 del/entry=xxxx ←
```

where `xxxx` is the entry number returned by the `sho que` command, 1767 in this instance.

3.5 Using the 24 position samplechanger

The 24 position samplechanger can be used either manually or under computer control. In both cases the initial setting-up procedure is the same. The samples are arranged on an endless chain and the can at the top of the samplechanger is viewed through the Perspex window (DO NOT CLEAN WITH SOLVENT!) by a webcam. The samples are tracked by the numbers on the clamping plate. This consists of six digits, a four digit number and a two digit checksum. Thus in *Figure 2.4* the number 003205 is visible, so this is: 0032 and (0 + 0 + 3 + 2 = 05). When a sample is in the beam, the can number is not visible but its counterpart 12 positions away is visible. Thus the first operation after loading the samples onto the samplechanger, see section 2.4, is to create a table of sample, can number and can number at the top when the sample is in the beam. An example is:

Can	**Top**	**Sample**
2507	707	0.5 mm cell H2O
2810	3609	1 mm cell H2O
2103	303	2 mm cell H2O
1203	404	Ti/NaAlH4 (fresh)
505	3508	Ti/NaAlH4 (after TPD)
1405	3306	Ti/NaAlH4 (after 10 bar H2)
202	1910	Ti/NaAlH4 (after 100 bar H2)
2709	3003	Sn/NaAlH4 (fresh)
1506	2204	Sn/NaAlH4 (after TPD)
2406	101	Sn/NaAlH4 (after 10 bar H2)
2911	2002	Sn/NaAlH4 (after 100 bar H2)
808	2608	n-propanol
707	2507	
3609	2810	
303	2103	
404	1203	
3508	505	
3306	1405	
1910	202	
3003	2709	
2204	1506	
101	2406	
2002	2911	
2608	808	

Note that not all the positions need to be filled, but pairs of cans must both have numbers.

After creating the table, the cables are removed and the samplechanger is then loaded into TOSCA using the crane, this must be done by the local contact. (**Note: the samplechanger must be bolted down before evacuating the IVC**). After installation, the main control cable, the Ethernet cable and the power supply for the webcam and the thermometer cable are reconnected. *Figure 3.3* shows the samplechanger installed in TOSCA and the important points highlighted.

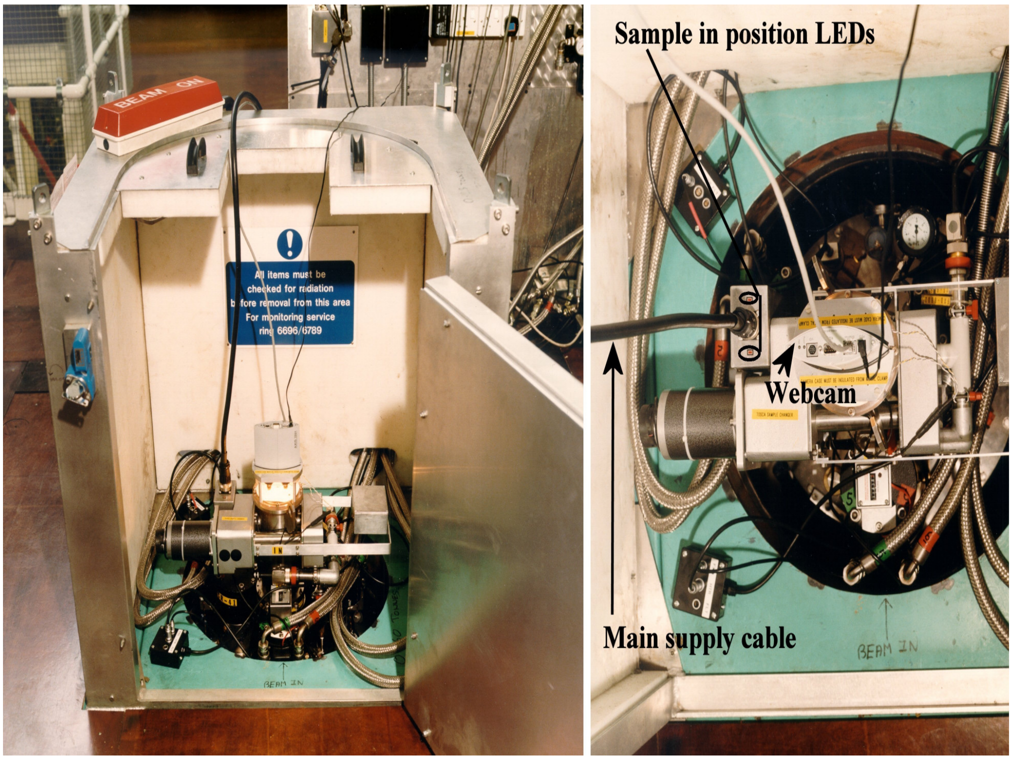
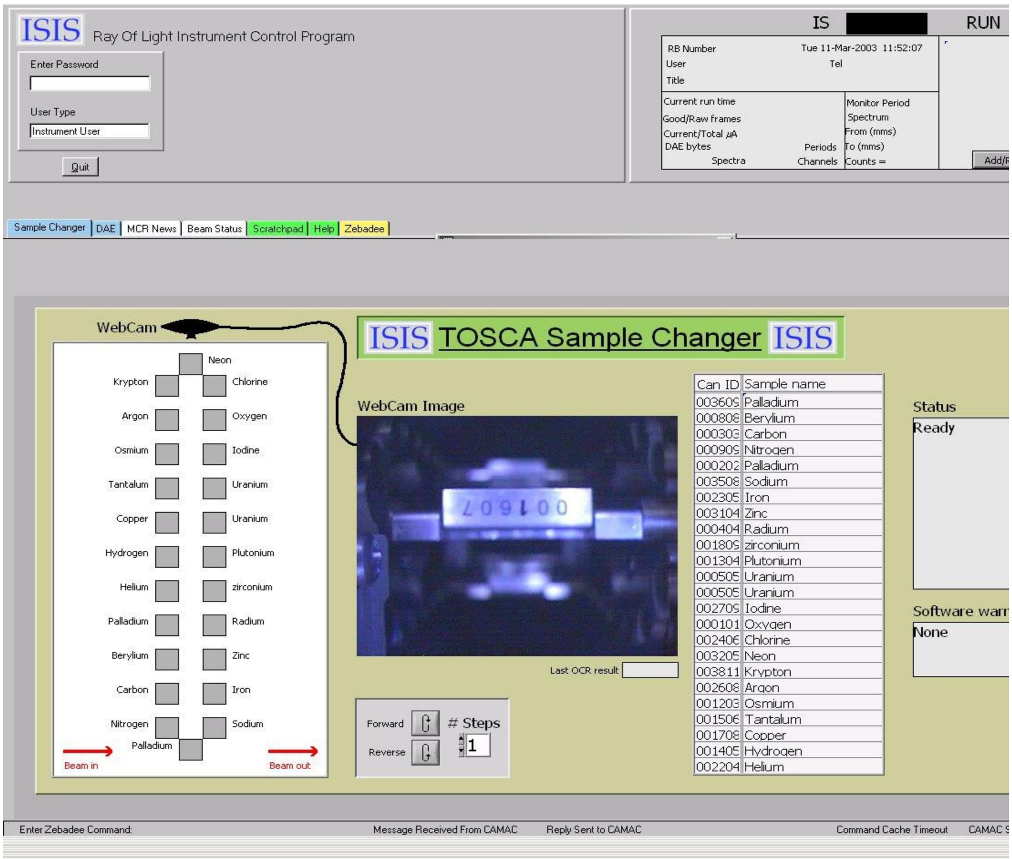

BEAM ON
All items must be checked for radiation before removal from this area
For monitoring service ring 6696/6789
BEAM IN
Sample in position LEDs
Webcam
Main supply cable
BEAM IN

Figure 3.3: The samplechanger installed in TOSCA.

Before setting the interlocks it is wise to check that everything is working! The lights immediately below the webcam should be on and the "sample in lock" light on the controller, *Figure 2.5*, and on the samplechanger, (indicated on the right-hand side of *Figure 3.3)*, should be illuminated. The webcam should also be working. This can be viewed in the TOSCA cabin using the Ray-of-Light programme on the PC next to the VMS screen (see later) or at: http://eye2.nd.rl.ac.uk/java on the general use PC. (It should be listed under Favourites in Internet Explorer).

The chain of command is: VMS → Ray-of-Light → controller → samplechanger. The samplechanger can be operated from any point along the chain, although full automation is only possible from VMS. The Ray-of-Light interface is shown in *Figure 3.4*.


ISIS Ray Of Light Instrument Control Program
Enter Password
User Type
Quit
Sample Changer
DAE
MCR News
Beam Status
Scratchpad
Help
Zebadee
WebCam
Neon
Krypton
Chlorine
Argon
Oxygen
Osmium
Iodine
Tantalum
Uranium
Copper
Uranium
Hydrogen
Plutonium
Helium
zirconium
Palladium
Radium
Berylium
Zinc
Carbon
Iron
Nitrogen
Sodium
Palladium
Beam in
Beam out
ISIS TOSCA Sample Changer ISIS
WebCam Image
001607
Last OCR result
Forward
Reverse
# Steps
Add/R
Enter Zebadee Command:
Message Received From CAMAC
Reply Sent to CAMAC
Command Cache Timeout
CAMAC S

User Type: Instrument User

IS [redacted] RUN

RB Number Tue 11-Mar-2003 11:52:07
User Tel
Title

Current run time
Good/Raw frames
Current/Total μA
DAE bytes Periods
Spectra Channels

Monitor Period
Spectrum
From (mms)
To (mms)
Counts =

Can ID	Sample name
003609	Palladium
000808	Berylium
000303	Carbon
000909	Nitrogen
000202	Palladium
003508	Sodium
002305	Iron
003104	Zinc
000404	Radium
001809	zirconium
001304	Plutonium
000505	Uranium
000505	Uranium
002709	Iodine
000101	Oxygen
002406	Chlorine
003205	Neon
003811	Krypton
002608	Argon
001203	Osmium
001506	Tantalum
001708	Copper
001405	Hydrogen
002204	Helium

Status: Ready

Software warn: None

# Steps: 1

Figure 3.4: The Ray-of-Light interface.

When the control system for the samplechanger was designed it was intended that it would be run exclusively from the Ray-of-Light interface (a GUI on top of the LabView control programme). Ultimately, this will happen when TOSCA is run from a PC rather than VMS. In practice, it is better to run the samplechanger from VMS and use Ray-of-Light simply as a connection in the chain. However, for the VMS commands to work, Ray-of-Light must be operating correctly. When the main supply cable is detached from the samplechanger Ray-of-Light will often hang. This is noticeable because the webcam view is not correct. It is re-started by pressing "QUIT" in the top left of the screen, the programme will exit and automatically re-start. This takes a couple of minutes.

The samplechanger can be moved by using the buttons in the lower centre of the screen. If you change the number of steps (=positions), labelled #Steps, then reset it to 1 afterwards because VMS uses the value set on the screen.

In theory, going forwards or backwards should work equally well. Experience shows that going backwards is when the samplechanger is most likely to jam, the only cure is to remove it from TOSCA, warm it to room temperature and put it back in the instrument. This can result in anything up to 12 hours lost time. You have been warned!

The samplechanger is run from VMS at the command level (where, END, BEGIN etc… are done), not in GENIE. The commands are:

next_sample	moves forward number of samples set in "#Steps"
prev_sample	moves back number of samples set in "#Steps"

prev_sample is not recommended as explained earlier. To ensure reproducible positioning, it moves back two positions, then forward one so that the limit switches on the samplechanger are always struck from the same direction. The commands are treated the same as any other VMS command, so can be included in a command file as usual.

4. DATA ANALYSIS AND VISUALISATION.

Several programs and utilities exist to help you analyse your data including the GAUS least squares fitting program. TOSCA is unusual in that for most cases the output from the automatic analysis program is all that is required. However, if desired, the raw time-of-flight data can be analysed independently. If you are doing data analysis in your own directory make sure that you are using the TOSCA GENIEINIT .COM. Please do not log onto TOSCA and start GENIE as this seriously slows the system; use a different computer instead e.g. ISISA.

4.1 GENIE

GENIE is the language used at ISIS for data manipulation. A full description of which, is available in the PUNCH user manual, a copy of which resides in the TOSCA cabin. Additional copies may be obtained from the computer support office.

If you are in the TOSCA cabin the GENIE window on screen two should already be opened. If there is no GENIE window or if GENIE crashes (rare but not unknown!) it is (re)started by typing "GENIE" (upper or lower case are both OK since VMS is case insensitive) in a DECTERM window (see section 3 and *Figure 3.1* for how to start a window). This will create an additional window that can be re-sized using the mouse. By default GENIE only uses the current screen, it does not scroll and allow you to use the scroll bar on the right of the window. This is inconvenient and can be corrected by clicking on the word "COMMANDS" at the top of the window and selecting the last item in the list "RESET TERMINAL".

If you are not in the cabin and are attempting to work on TOSCA data elsewhere, *e.g.* in the DAC, the procedure is more complicated and you should consult your local contact before starting.

When GENIE starts, a page will scroll past which includes each specialised function available and the command needed to utilise them. For reference a copy of this can be found in section 7.2. Typing the command:

```
sho sym ←
```

will type the list.

On TOSCA, GENIE is divided into 16 workspaces, w1 – w16. The data in GENIE is completely volatile and does not alter the original data on the TOSCA disk. Thus no operation, up to and including crashing GENIE, will result in loss of data.

4.1.1 Looking at analysed files

For each run, three analysed (i.e. as intensity vs. energy transfer) files are stored. These are the: the summed back scattering detectors, the summed forward scattering detectors and the sum of the two sets of detectors (back + forward). These are called:

trsl	sum of back and forward detectors
trslba	backscattering detectors
trslfo	forward scattering detectors

To read in an analysed file type:

```
»r wl [tosca.user]trslxxxx.ana ←
»r wl [tosca.user]trslbaxxxx.ana ←
»r wl [tosca.user]trslfoxxxx.ana ←
```

For the total, back or forward scattering detectors respectively, where xxxx is the run number. For TOSCA-1 (run numbers 1 - 2134)) there are only backscattering spectra, so only the first command is applicable. For TFXA spectra use:

```
»r wl [tfxa.user]trslxxxx.ana ←
```

To display a spectrum (assumed stored in workspace 1) type:

```
»d wl ←
```

This will plot the spectrum in the GENIE graphics window. The range of data displayed may be specified:

```
»d wl 50 100 0.1 0.3 ←
```

This plots workspace 1 between 50 and 100 (x units) and 0.1 and 0.3 (y units). GENIE assumes that the two numbers following the workspace number are x values; to specify a y range, an x range must be given first.

4.1.2 Types of plot

As well as the histogram plot, it is also possible to plot the data as points, line plots or error bars. To change the type of plotting, type:

```
»d/l ← For a line plot
```

```
»d/h ← For a histogram plot
```

```
»d/e ← For error bars
```

```
»d/m ← For the data points
```

The display defaults to the last of these options entered.

4.1.3 Overlaying spectra

To overlay spectra one on top of another you can type:

```
»p w? ←
```

Where ? is the number of the workspace to be added to the current graphics window. This is useful for comparing accurately two or more spectra. A useful device is to display the data using the d command in a histogram format and then to overlay the error bars by using:

```
»p/e ←
```

4.1.4 Binning and rebinning

The bin size represents the number of adjacent points averaged for each data point. So a binning of 1 *(i.e.* no binning) has a high accuracy, but may also have high noise levels. A numerically larger binning will give reduced noise, but the resolution will be degraded, thus binning acts as a crude type of smoothing. The advantage is that the data in the workspace is not permanently changed. To alter the binning, type:

```
»a b x ←
```

Where x (integer only) represents the binning number, usually between 1 and 10.

The GENIE command "rebin" allows different portions of the workspace to be averaged to different extents (unlike binning which operates on the whole workspace), but permanently changes the data in the workspace. Thus the best method is to copy the data to another workspace by *e.g.*:

```
»w1 = w2 ←
```

and then to experiment on the second workspace. There are two forms to the command:

```
»reb w2 16 (2) 4000 ←
```

```
»reb w2 16 [.02] 200 [.005] 4000 ←
```

In form 1 with ( ) brackets the value inside the brackets is in x units. So for a spectrum in wavenumbers, the first command will rebin the data into 2 cm^{-1} intervals between 16 and 4000 cm^{-1}. Rebin truncates to the limits given. Thus if the upper limit was 2000 cm^{-1} then the data would only be retained between 16 and 2000 cm^{-1}. Note that multiple ranges are possible, as shown in the second example.

In form 2 with [ ] brackets the value inside the brackets is related to the size of the time bins in the raw time-of-flight data. When the data is transformed from time-of-flight to energy transfer a value of 0.005 is used across the entire spectrum. Thus in form 2 the spectrum is being rebinned in "4’s" (0.02 = 4 x 0.005) between 16 and 200 and in "1’s" between 200 and 4000 cm^{-1}. This is a particularly useful form of the command since at low energies there are many more data points than can be justified by the instrument’s resolution function. Thus the data can be rebinned to improve the signal-to-noise without degrading the resolution.

4.1.5 Hard copies

To obtain a hard copy of the current data that is displayed in the GENIE graphics window, type:

```
»lpr ←
```

A list of printers is then given and you are prompted for which printer to use (See *Table 4.1*). Just type the printer number and a postscript file will be automatically created and sent to the nominated printer.

Laser printers	**Location**

LASER 0	Computer support office, R3.
LASER 1	Coffee room, R3.
LASER 2	DAC, R55.
LASER 17	TOSCA cabin

Table 4.1: A list of the normally used printer devices.

A procedure that will always work but is somewhat more long-winded is to type:

```
»k/h ←
```

This carries out a screen dump of the GENIE display window and creates a postscript file called DEC_POSTSCRIPT.DAT, in the directory you are currently in. To print this file from GENIE:

```
»j "plaserx dec_postscript.dat" ←
```

Where **x** is the number of the laser printer (see table 4.1). Remember to change the disk/directory name if your file is elsewhere. These files are purged frequently so it is inadvisable to do too many at any one time.

4.1.6 Using the cursor

Should you wish to find the exact co-ordinates of a peak for example you can type:

```
»c ←
```

A 'cross-hair' will appear in the GENIE graphics window. This can be positioned using the mouse. When in the correct place click the left button and a menu will appear, select the desired option. To exit from the cursor, it is necessary to choose the "EXIT" option from the menu. By default, values and text are printed vertically at the cursor position. For annotation of a plot this is inconvenient, if

```
»c/h ←
```

is used then the output is horizontal.

4.1.7 Useful functions in GENIE

As well as the built-in functions of GENIE, there are some routines that are specific to TOSCA that are useful to know about: For most of the

programmes, when prompted for a workspace, type:
w? (1 ≤ ? ≤ 16). Exceptions will be noted.

QR4

This is to have a look at the spectrum as it is being recorded. Type:

```
»qr4 ←
```

This will result in an analysed spectrum **that is not saved.** The output of **QR4** is placed in workspace w5. The back scattering detectors are in w7, the forward in w9 and the sum in w5. Note that workspaces w1 - w9 are overwritten during the analysis.

FR4

This is the main TOSCA data analysis program. It is normally only used if data outside the usual range (2 - 500 meV, 16 - 4000 cm^{-1}) is required or if saved (.SAV) or co-added files are to be analysed or when the batch file does not run or when the .ANA file has been corrupted and the data has to be re-analysed. (For co-adding sequential runs there is a version of FR4 called SR4, see next topic). Note that the . RAW file is unchanged by FR4 in any of its manifestations.

The use of a fixed final energy on TOSCA means that each energy (ω) is associated with a unique value of momentum transfer Q. A second consequence is that Q is only weakly dependent on the scattering angle, thus for the small angles subtended by the detector banks, there is no variation in Q across the detector bank. This means that the analysis of the raw time-of-flight data on TOSCA is straightforward. In essence, it consists of normalising each detector spectrum to the incident monitor spectrum, conversion to energy transfer (in meV) and summing the detectors to give a single spectrum. This process is sufficiently routine that it is carried out automatically by a batch file each time a run is ended and uses the raw time-of-flight data file (.RAW) to generate the files TRSLxxx.ANA, TRSLFOxxx.ANA and TRSLBAxxx.ANA in the directory TOSCA$DISKO:[TOSCA.USER] a few minutes later.

To run FR4 type:

```
»fr4 ←
```

The program asks for a number of inputs. In the order in which they are required these are:

File extension:
1 for . RAW original data
2 for . SAV files saved during a run

3 for . SUM co-added data files

Run number.

Energy binning choice and a value:
1 for constant ΔE/E.
2 for constant ΔE.

The raw time-of-flight data is collected in bins of equal width, thus as the energy transfer increases, there are fewer time bins in a given energy width. For most cases ΔE/E is the better choice since it better matches the resolution function of the instrument and ensures that there are sufficient data points at each energy to correctly define the resolution. ΔE is ***not*** the resolution, it is the width *in energy* of a time bin. The required value can be calculated from:

$$\Delta E/E \approx \sqrt{}\ 0.0002\ E_{max}$$

where E_{max} is the highest energy (in meV) required in the spectrum. For the standard range 2 - 500 meV a value of 0.005 is appropriate.

The energy range to analyse.

This is usually 3.5 - 500 meV (30 - 4000 cm^{-1}). Care is needed since there is an interplay between the type and size of the energy binning and the energy range. Note that if the highest energy is too large or the value given for ΔE/E is too small, then at some energy, there will be less than one time bin per energy element which results in a computer error.

Type of output:
1 for double differential
2 for S(Q,w) **(usual choice)**
3 for both
4 for neither

Whether to exclude any detectors. Only in exceptional circumstances would this be required *e.g.* single crystal studies. For most samples the detectors are evenly illuminated, thus excluding detectors results in a reduction in signal-to-noise. If detectors are to be excluded type 1 and then either 1 (include) or 0 (exclude) for each detector in turn

FR4 then analyses the data, like QR4 it uses workspaces w1 - w9 and puts the data in w5, w7 and w9 at the end of the analysis. It also writes the analysed data to:

```
tosca$disk0:[tosca.user]trslxxx.ana
tosca$disk0:[tosca.user]trslbaxxx.ana
tosca$disk0:[tosca.user]trslfoxxx.ana
```

so it may be read in subsequently.

Thus to analyse data using the standard conditions the series of inputs are:

```
1 ←
1 ←
.005 ←          Value must be in this format, 0.005 will
                  not  work
2 ←
500 ←
2 ←
2 ←
```

SR4

This program analyses spectra that were collected sequentially and co-adds the result. The co-addition is performed *before* the data is converted to energy transfer since this results in slightly better statistics. The operation is the same as for FR4 except that the user is prompted for the number of spectra and the first run number. It writes the analysed data to:

```
tosca$disk0:[tosca.user]tsumslxxx.ana
tosca$disk0:[tosca.user]tsumslbaxxx.ana
tosca$disk0:[tosca.user]tsmslfoxxx.ana
```

where xxxx is the run number of the first of the consecutive files.

TEMP_PLOT and TEMP_PLOT_CURRENT

These programs plot the temperature vs time for an old run and the current run respectively. Type

```
»tp ←
```

or

```
»tpc ←
```

Both programs are run in the same way and are very similar. The only difference is that after starting tp you are asked for the run number and then asked to give the start date and time, whereas tpc immediately asks for the start date and time. The simplest method is to accept the defaults, which is just a return. If you want to input values then these must be in the format:

```
xx-mon-year hr:min:sec
```

e.g.

```
12-jul-1995 09:45:00 ←
```

The space between the year and the time is essential. You are then asked for the finish time in the same format. When prompted:

```
»Give Se block name
```

Type `temp` for the sample temperature history or `temp1` for the cryostat temperature history. You are then prompted for the temperature units (K or C) and for which log column, the default is 3 and this should be used. The program then extracts the relevant data from the temperature log. *This may take several minutes so be patient!* Eventually, it comes up with the message:

```
»Ok. Toggle mode to point plotting and d/l wl
```

"Toggle mode" switches between using the edge of data bins and the centres. Unless there are very few data points available, there is no visible difference between plots using the two modes (see "Toggle" in the GENIE manual for details). The data is stored in workspace 1 and can be treated as normal. Note that because it was generated in GENIE, it is completely volatile. It can saved as ASCII data using B2A or as binary data using the GENIE "write" command (see GENIE manual for details).

B2A

This program converts a binary file (located in a workspace) into a three column ASCII file (x, y and error) which is suitable for input to aCLIMAX, CLIMAX or to a spreadsheet. Type:

```
»b2a ←
```

you will be prompted for the workspace, w?, and a filename. Note that b2a automatically adds .DAT to the name you give it. You are then prompted for the first and last x values (although it asks for the values in meV, it actually uses whatever the workspace x units are; cm^{-1}, Å, Å^{-1}).

LOAD_B2A

This program inputs an ASCII file to GENIE. It assumes the same format as that created by B2A i.e. first line is the title, second line is the units and then three space separated columns, x, y, error. To use it type:

```
>>load w? <filename> [tosca.command]load_b2a
```

The syntax is:

```
load w? [tosca.command]load_b2a
```

e.g.

```
load w1 1butoh.dat [tosca.command]load_b2a
```

If the ASCII file is not in the same directory from which GENIE was started (usually TOSCA$DISK0:[TOSCA]) then <filename> must include the complete path.

CONVERT

This program coverts the x-axis scale from meV to wavenumbers. Type:

```
»con ←
```

you will be prompted for the workspace, w?, to be converted and then for the output workspace. This can be the same or different to the input one.

STRETCH

This program is similar to CONVERT but the x-axis multiplicative factor is provided by the user. This is useful for going from e.g. meV to THz, cm^{-1} to meV and for scaling by $1/\sqrt{2}$ to simulate the expected spectrum for a deuterated sample from that of the protonated one. Type

```
»str ←
```

you will be prompted for the workspace, w?, to be converted, then for the output workspace and then for the scaling factor, this can be any real number (+ or -).

GAUS

This gives access to a program that performs a least-squares fit of a sum of Gaussian lineshapes to the experimental data. Type:

```
»gaus ←
```

For more information see the FRILLS manual.

SMOOTH

This creates a smoothed spectrum using the Savitsky-Golay method. This programme overwrites the input workspace, so it is sensible to copy the data to another workspace first. To obtain the best results it is usually necessary to have several attempts. The user is prompted for the workspace number, in this case enter the number, not w? i.e. 12 not w12. The % smoothing is then asked for, generally 1 - 10% is optimal. The order of the polynomial is then requested, 0, 2, 4. The severity of the smoothing is 0 > 2 >4. Type (user input in bold):

```
>>smo ←
```

The screen returns:

```
>> @TOSCA$DISK0:[TOSCA.COMMAND]SMOOGEN>>
>> Data smoother using Savitzky-Golay method
>>
>> ENTER WORKSPACE: 1
```

For the workspace type the workspace number ? ***not*** w?. The screen returns:

```
>> Workspace to be smoothed: 1
>> SMOOTHING (%): 10
```

This controls the degree of smoothing, any integer 1-100 is usuable but 10 is a reasonable compromise between noise reduction and loss of resolution. The screen returns:

```
>> Percentage of smoothing: 10
>> ENTER ORDER (0, 2, 4): 4
```

This also affects the quality of the ouput. 4 works well for smoothing a background. The screen returns:

```
>> Smoothing polynomial order: 4
>> TR W1 TOSCA$DISK0:[TOSCA.COMMAND]SMOOGEN.EXE W1
No OF DATA POINTS= 1109
No OF WINDOW POINTS= 111
POL. ORDER= 4
```

If the result is not to your liking, copy the original data to another workspace and experiment with the % smoothing and the polynomial degree until you get something you do like.

SPIKE

Occasionally, a spectrum will contain a spike in the data caused by a glitch in the detector or DAE electronics. There are two ways of dealing with this: convert the file to ASCII using B2A, edit out the offending points and then put it back into GENIE with LOAD_B2A. Alternatively, it can be done using SPIKE, which draws a straight line between two user-defined points. Display the spectrum and using the cursor, note the edges of the region to be removed. The sequence of commands is (user input in bold):

```
>> spike
>> @TOSCA$DISK0:[TOSCA.COMMAND]SPIKE.COM
>>
>> Getting rid of spikes >>
>> ENTER INPUT WORKSPACE NUMBER: 1
>> Workspace to be cured: 1
>> ENTER OUTPUT WORKSPACE NUMBER: 5
>> Cured workspace : 5
>> first x-axis point: 1355
>> First x-axis point
>> last x-axis point: 1390
>> Last x-axis point
>> W5=W1 Load Workfile 1 into Workfile 5
>> TR W5 TOSCA$DISK0:[TOSCA.COMMAND]SPIKE.EXE W5 No
OF DATA POINTS= 996
```

COMP

This programme corrects for a misaligned sample. If the sample is not at the centre of the instrument, i.e. it is displaced toward or away from the moderator, this results in a shift in the spectral positions that increases linearly with increasing energy transfer. This manifests as the peaks in the forward and backscattering spectra appearing at different positions. The commonest cause is samples being mounted proud of the aluminium frame on the samplechanger. If the shift is small, then the peaks are broadened, if it is large the peaks may be doubled. It is good practice to always overlay the forward and backscattering spectra to check for a misalignment rather than just relying on the sum file. Fortunately, the shift in positions is symmetric about the true position. This programme uses the difference in position of a band between the forward and backscattering spectra to generate corrected spectra. Before running the programme use the cursor to determine the position of the same sharp peak in the forward and backscattering spectra The sequence of commands is (user input in bold):

```
>> comp
>> @TOSCA$DISK0:[TOSCA.COMMAND]COMP.COM >>
>> Compensating the back-scattering and >> forward-
scattering det. banks >>
>>
>> ENTER INPUT WORKSPACE NUMBER (BA): 1 >> BA
```

```
workspace to be compensated: 1
>> ENTER OUTPUT WORKSPACE NUMBER (BA): 5 >>
Compensated BA workspace: 5
>> ENTER INPUT WORKSPACE NUMBER (FO): 2 >> FO
workspace to be compensated: 2
>> ENTER OUTPUT WORKSPACE NUMBER (FO): 6 >>
Compensated FO workspace: 6
>> In meV (1) or cm^-1 (0) ?: 0
>> Energy units of the following w points >> BA w-
axis point: 1082
>> BA w-axis point
>> FO w-axis point: 1090
>> FO w-axis point
```

In this example the spectra to be corrected are in w1 and w2 and the corrected spectra are put in w5 and w6. Any workspaces may be used. The user is prompted for the input and output workspaces (number only, not w?) for the backscattering and forward scattering detectors, whether the units are meV or cm-1, and the position of the same peak in the forward and backscattering spectra.

DERIVATIVE

This calculates the derivative spectrum for a chosen workspace. Type:

```
»der ←
```

Then follow the on screen instructions. Note that it uses additional workspaces for intermediate steps so data may be overwritten.

4.1.8 Assigning files

It is occasionally necessary to look at the raw time-of-flight data from the individual detector tubes. To do this type:

```
»ass dae ←
```

for the current run or

```
»ass xxxx ←
```

where xxxx is the run number for a previous run. To display an individual spectrum:

```
»d s? ←
```

where ? is the tube number. Tubes 1 - 140 are the inelastic detectors, tube 141 is the monitor and tubes 146 - 149 are the diffraction detectors. To manipulate the data it must be put into a workspace. For example, to put spectrum 3 into workspace 1, type:

```
»w1 = s3 ←
```

Then display as described earlier.

To look at more than one tube use the multiplot command (see GENIE manual):

```
»mu s1>s56 1000 20000←
```

This will display the data in detectors 1 to 56 between 1000 and 20000 m s.

4.2 OPENGENIE

OPENGENIE is the successor to GENIE. It is more flexible (e.g. unlimited number of workspaces, able to handle two-dimensional data sets, command line recall by use of up-arrow, etc…). A HELP file is available on-line on the web at http://www.isis.rl.ac.uk/GenieReferenceManual/. The advantage for TOSCA is that it enables a QREHACK to be carried out in less than a minute rather than the five minutes or so required by GENIE.

To start OPENGENIE, create a DECTERM in window 3 and then type at the TOSCA prompt:

```
TOSCA>OPENGENIE←
```

A series of initialisation messages are printed followed by a *caveat emptor* notice. OPENGENIE is different from GENIE in that the number of workspaces is essentially unlimited and they can be called almost anything. However, for continuity with GENIE, workspaces will be called w1 to w16.

To look at the current run type:

```
»qrehack_fb←
```

The S(Q,ω) data is placed in w5 (forward scattering detectors) and w7 (backscattering detectors). To display the file type:

```
»d w5 50 100 0.1 0.3←
```

A PGPLOT window is created and the data displayed in it as shown in *Figure 4.1*. Thus the display command is the same as in GENIE. The p (overlay) command is also the same.

The default x axis of the display is meV, to convert to cm^{-1} type:

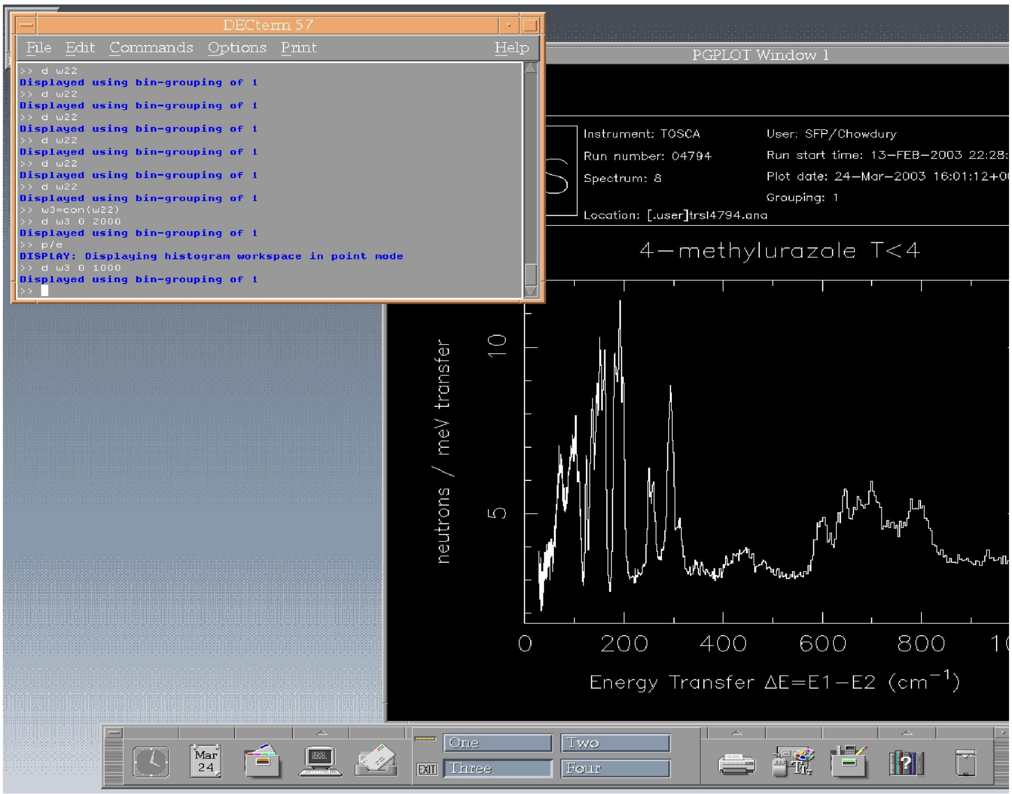
```
»w? = con(w5)←
```

where ? is any integer.

To change the binning type:

```
»a/b ?←
```

(note difference from GENIE which is a b ?)



DECterm 57
File Edit Commands Options Print Help
PGPLOT Window 1
neutrons / meV transfer
10
5
0
200
400
600
800
Energy Transfer ΔE=E1−E2 (cm⁻¹)
Mar 24
One
Two
Three
Four
EXIT

```
>> d w22
Displayed using bin-grouping of 1
>> d w22
Displayed using bin-grouping of 1
>> d w22
Displayed using bin-grouping of 1
>> d w22
Displayed using bin-grouping of 1
>> d w22
Displayed using bin-grouping of 1
>> d w22
Displayed using bin-grouping of 1
>> w3=con(w22)
>> d w3 0 2000
Displayed using bin-grouping of 1
>> p/e
DISPLAY: Displaying histogram workspace in point mode
>> d w3 0 1000
Displayed using bin-grouping of 1
>>
```

Instrument: TOSCA
Run number: 04794
Spectrum: 8
Location: [.user]trsl4794.ana
User: SFP/Chowdury
Run start time: 13-FEB-2003 22:28:
Plot date: 24-Mar-2003 16:01:12+0
Grouping: 1

4-methylurazole T<4

Figure 4.1: The OPENGENIE control window and display window.

To plot the spectrum the lpr command is used as in GENIE, type:

```
»lpr ←
```

This will print the list of printers given in *Table 4.1* and ask which printer to use. Just type the printer number and a postscript file will be automatically created and sent to the nominated printer.

To look at previously analysed files type:

```
»w?=get(1,"[tosca.user]trslxxxx.ana")
»w?=get(1,"[tosca.user]trslbaxxxx.ana")
»w?=get(1,"[tosca.user]trslfoxxxx.ana")
```

? is any integer and xxxx is the run number.

To use the cursor type "`cursor`" at the prompt rather than just "`c`" as in GENIE.

Further TOSCA utilities will be added as time, enthusiasm and demand enable!

---

5. THE HARDWARE ON TOSCA

The purpose of this section is to supply practical information on where things are on TOSCA and how they work. It also supplies information on what the user can attempt without the risk of damaging the instrument and what should be left to the instrument scientist.

5.1 The Instrument

5.1.1 The Vacuum

TOSCA has two separate vacuum systems; the sample tank that contains the cryostat and a second vacuum system that houses the beryllium filters. Both of these are pumped with turbomolecular pumps to a cryogenic vacuum of better than 10^{-5} mbar. The sample and beryllium filter tanks on TOSCA are separated by aluminium windows.

The spectrometer vacuum pumps are located beneath the spectrometer, access is interlocked. The vacuum gauges (see *Figure 5.1*) and controls for the pumps are in the electronics cabinet on the mezzanine level in front of the TOSCA services panel on the side furthest from the target station (see *Figure 2.3*) . The upper panel is for the beryllium filter and the lower panel is for the sample environment tank. The display is in millibar. You will NOT normally touch any of this equipment, however, it is good practice to check the pressure in the two systems once or twice a day.

If the pressure is greater than 1 x 10^{-5} mbar you should inform your local contact immediately.

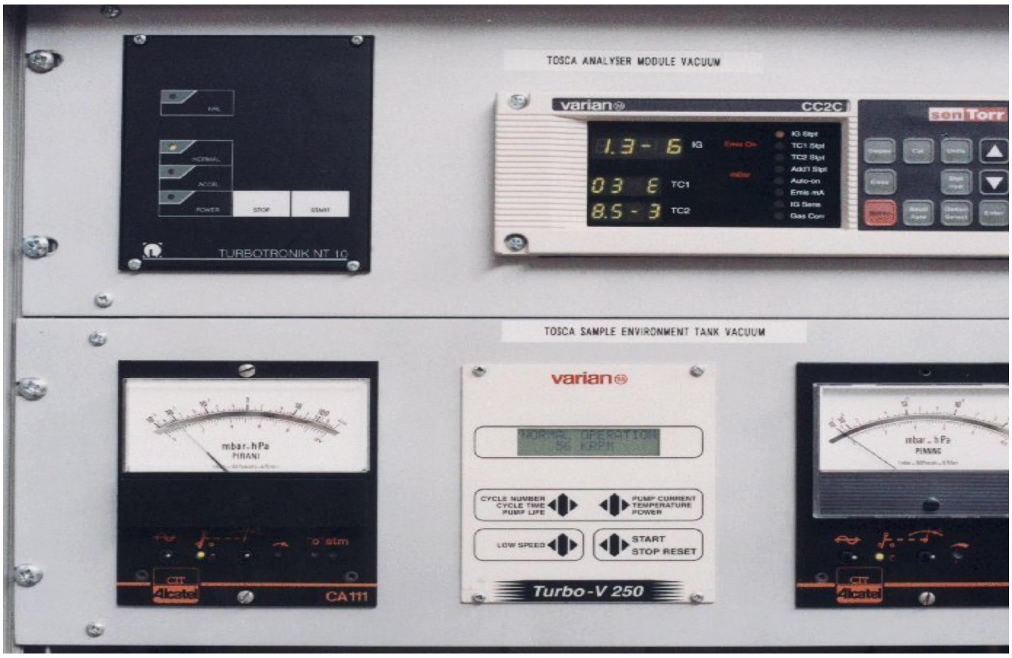

TOSCA ANALYSER MODULE VACUUM
varian
CC2C
senTorr
1.3 - 6
IG
03 E
TC1
8.5 - 3
TC2
TURBOTRONIK NT 10
NORMAL
ACCEL
POWER
STOP
START
TOSCA SAMPLE ENVIRONMENT TANK VACUUM
mbar - hPa
PIRANI
Alcatel
CA111
varian
NORMAL OPERATION
56 KRPM
CYCLE NUMBER
CYCLE TIME
PUMP LIFE
PUMP CURRENT
TEMPERATURE
POWER
LOW SPEED
START
STOP RESET
Turbo-V 250
mbar - hPa
PENNING
Alcatel

Figure 5.1. The vacuum gauges on TOSCA

5.1.2 The Beryllium Filters

The transmission of a beryllium filter is approximately doubled by cooling below ~100K. On TOSCA each Be filter is attached to a single stage CCR that cools it below 50K. The ten CCR heads are driven by five compressors that are located outside the hall, adjacent to the nearest exit from R55 (see *Figure 5.2*). The temperature of the heads is measured by Pt resistance thermometers. The output of these is displayed in the bank of ten readouts in the section of the TOSCA cabin that holds the electronics racks (see *Figure 5.3*). Each of the filters should be below 50K, there is an alarm signal (the red LED on the panel) if the temperature exceeds this. Again it is a useful precaution to check the temperatures of the Be filters once or twice a day. If a filter is above 50K check that all the compressors are operating and then inform your local contact.

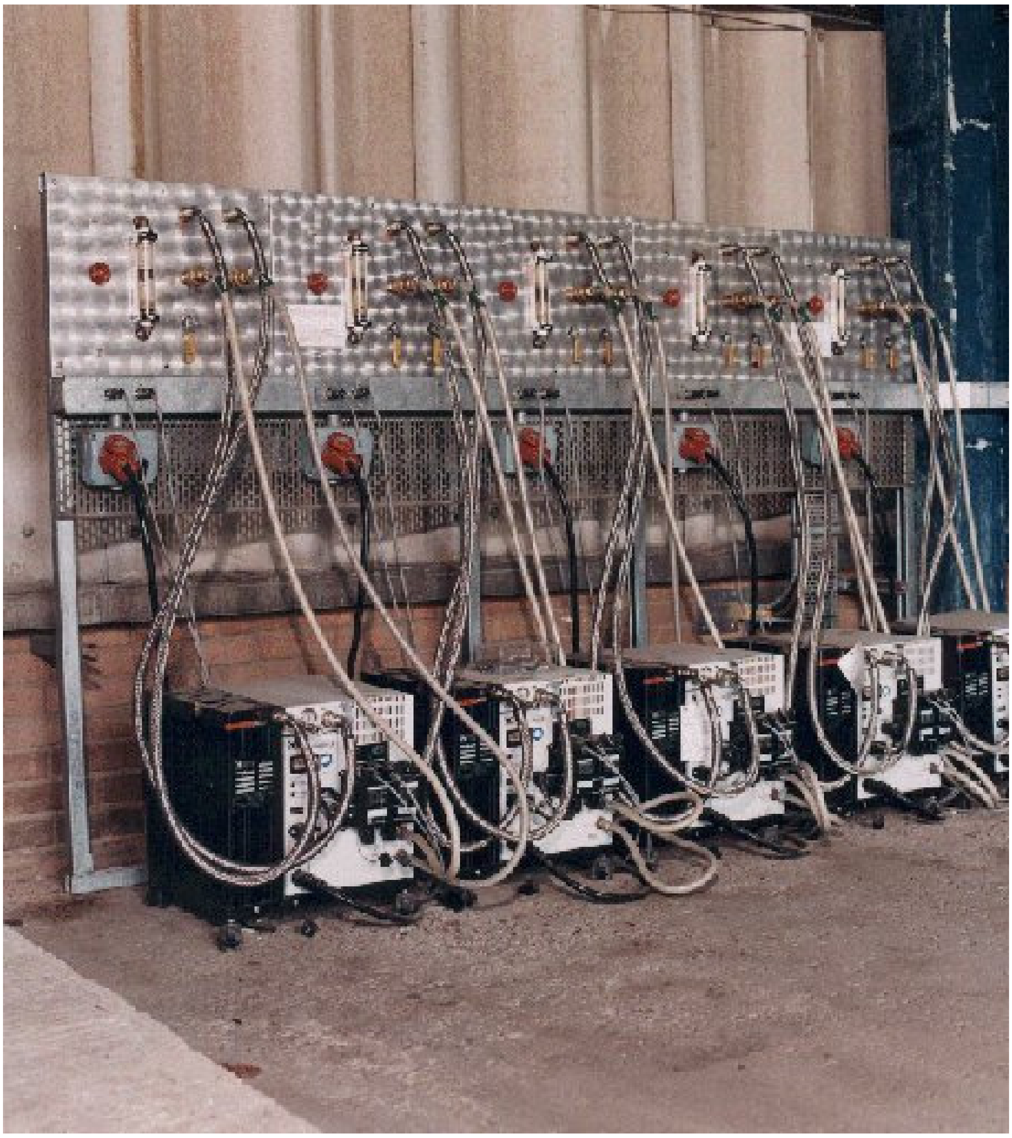


Figure 5.2: The compressors for the Be filters outside R55.

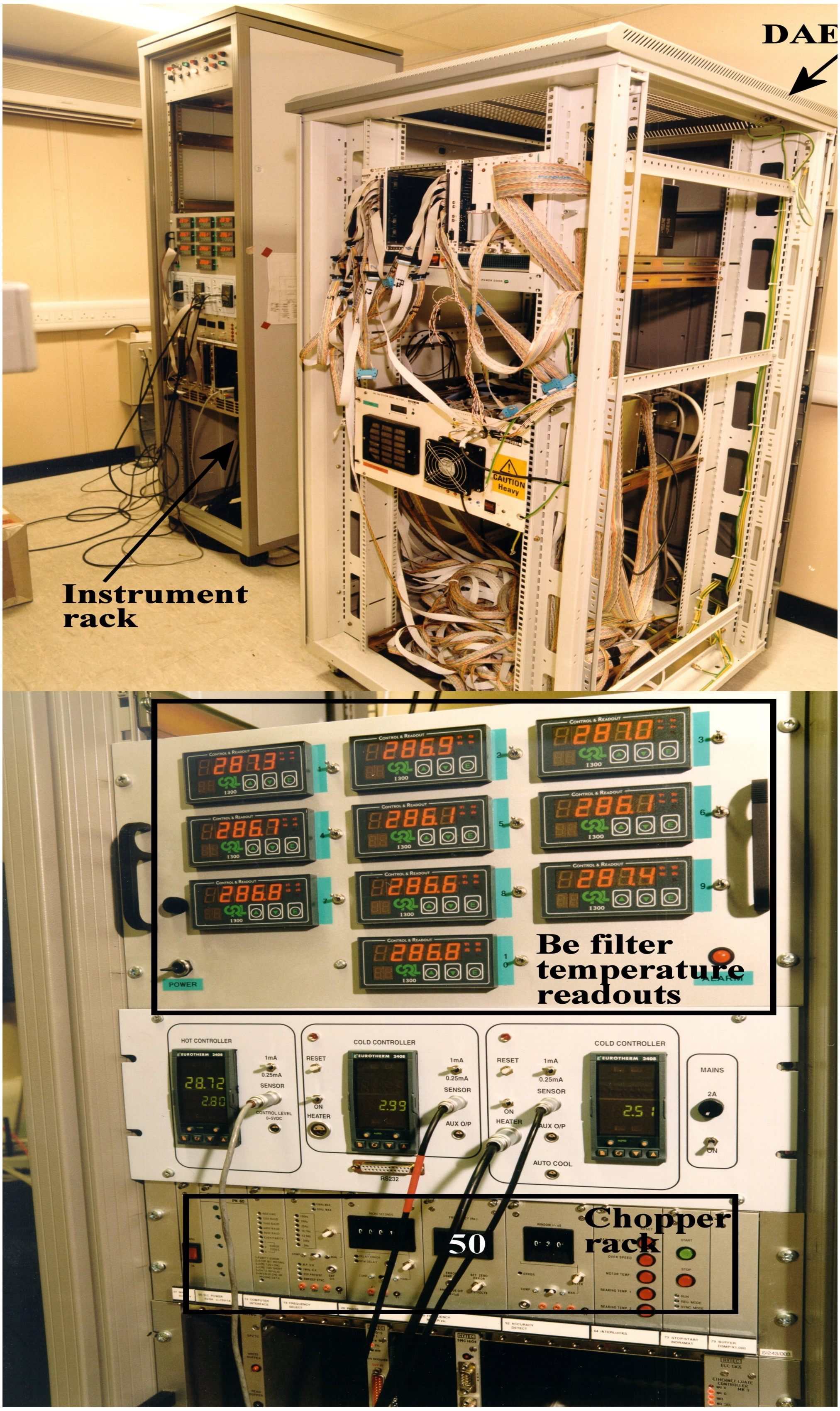

DAE
Instrument rack
Be filter temperature readouts
HOT CONTROLLER
COLD CONTROLLER
COLD CONTROLLER
MAINS
Chopper rack
50

Figure 5.3: (a) The section of the TOSCA cabin that holds the electronics racks showing the key elements. (b) The Be filter temperature readouts and the chopper controls. Note "50" in the

middle of it.

5.2 The top loading CCR

The major piece of sample environment equipment available on TOSCA is the top loading CCR. This is designed to be compatible with the standard ISIS centresticks, thus the distance from the underside of the flange to the beam centre is 1165 mm and the bore is 100 mm. The advantage of this system is that the sample can be changed without having to warm the cryostat to room temperature. It is cooled by three CCR heads, one single-stage compressor cools the outer (thermal) radiation shield to ~40K and two, two-stage heads cool the inner vacuum vessel to ~12K. The sample is cooled by helium exchange gas in this vessel. As described in section 3, the cryostat and sample temperature's are displayed on the Dashboard. The CCR heads are driven by the two compressors located inside the hall on the outer wall on the ground floor of R55, see *Figure 2.15*. If the sample or cryostat temperature starts to rise unexpectedly you should:

- Check whether the sample or cryostat is being heated.
- Check that there is He exchange gas in the cryostat, (there is a gauge on top of the cryostat, see *Figure 2.13*.).
- Check that both compressors are on.

If in doubt contact your local contact.

5.3 The Nimonic chopper

The Nimonic chopper on TOSCA at 9.5 m has two purposes; it removes the γ-flash (γ-rays and unmoderated fast neutrons from the proton beam hitting the target) which only contribute to background. There is a piece of B_4C on the leading edge of the chopper that removes slow neutrons from the neutron pulse preventing frame overlap. The chopper is phased to remove as many high energy neutrons as possible. The main electronics and power supply are on the ground floor underneath the TOSCA cabin and the controls are in the rack in the partioned part of the cabin, see Figure 5.3b. The rotation rate should be 50 Hz and this is indicated by the digital display on the control rack. *If this reads anything other than 50 inform your local contact.* Neither the phase nor the rotation rate should be altered by the user.

---

6. THE VITAL STUFF

6.1. Beam off

You can check if the beam is off in a number of ways; the beam current displays at both ends of the experimental hall will read "BEAM OFF", the Dashboard display will read zero current and your data will not improve with time! You can get information on what has happened and how long the beam will be off by typing in the TOSCA Control window:

```
TOSCA> ISISNEWS C ←
```

You exit by pressing "Control Y".

The beam statistics can be checked via the internet. At http://www.isis.rl.ac.uk there is a link to a page containing information about the beam status. From here graphs of the beam current can be obtained. There is a link to the MCR message board containing repair information and estimated fix times.

6.2. A final checklist

Before you walk out of the cabin for a quiet night in the pub, quickly go through the following checklist.

- Interlocks complete
- Shutter open
- Vacuum good
- Be filter temperature's <50K
- Command file, e.g. for temperature changes, edited, stored and running

Dashboard shows "RUNNING" (or possibly "WAITING" if using a command file).

6.3 Useful phone numbers

In the event of any problems with the instrument, computing or sample environment your first point of contact should be your **local contact.** If they are unavailable, then you should contact Stewart Parker, Timmy

Ramirez-Cuesta or John Tomkinson. The home numbers can be used in the case of problems in the evening, but please not after 11 o'clock, except for dire emergencies. The Main Control room is manned at all hours and they can also be contacted if you have a problem, ext. 6789. If you have queries about accommodation, claims or transport contact the University Liaison Office (ULS) inside working hours, ext. 5592, email: isisuo@rl.ac.uk.

To dial an office extension from outside RAL 01235 44+extension number
To make an external call from a RAL phone normal number

Other useful numbers:

Emergency Fire or Ambulance	2222
Main Control Room	6789
Health Physics	6696
University Liaison Office	5592

6.4 Safety summary

Before you start your experiment please make sure that:

- You have registered with the University Liaison Office (ULS) in R3, or in the Main Control Room (MCR) if you arrive outside working hours. If you are a new user you will be issued with safety instructions, read them. You must also watch the safety video and sign the yellow card.
- You have picked up a film badge from the Health Physics Office opposite the MCR and a swipe card from the MCR.
- You have picked up and read the sample record sheet from the Data Acquisition Centre (DAC) and that you understand the sample handling instructions. This sheet is to be displayed on the instrument during the experiment.

The full safety instructions are to be found in the literature given out by the ULS. However, the salient points concerning the instrument are summarised here.

After the experiment the sample should be monitored. If the radiation is:

- Greater than 75 µSv (%beta; or γ). The ISIS duty officer (ext. 6789) must be informed to supervise the removal of the sample. Any operation concerning the sample must also be supervised by the duty officer.
- Greater than 10 µSv. The sample can be removed and stored in the active sample cabinet. However, any operation that requires the sample can to be opened must be done in an active glove box.

- Less than 10 µSv. The sample can be handled normally, using good laboratory practice.

After the completion of the experiment the sample can and sample should be placed in the active sample cabinet in a suitable container with a copy of the sample record sheet.

If it is necessary to transport an irradiated sample off-site, documentation must be obtained from the health physics office. Do not take the sample to them, they will come down to the instrument. Preparing the documentation will take some time so ask for this well in advance of departure.

ISIS conforms to COSSH regulations. Any chemical process or procedure that involves chemicals, must be assessed beforehand by ISIS Safety personnel.

If you have any safety concerns ask your local contact or ring the Main Control Room.

6.5. Eating and Drinking

6.5.1. RAL Opening Hours

R22 Restaurant
The times below were correct at 26/3/03. However... things change! You are advised to check:
http://www-internal.clrc.ac.uk/catering/restaurant/Opening_TimesoftheRestaurant.html
for the most recent information.

		ISIS running	**ISIS shutdown**
Monday to Friday	Breakfast	7.30 - 9.00	7.30 - 9.00
	Lunch	11.45-13.45	11.45-13.45
	Light meal	17.00 - 18.00	17.00 - 17.30
	Restaurant	18.15 - 20.30	17.30 - 19.15
Weekend	Breakfast	8.00 - 9.00	8.00 - 9.00
	Lunch	12.00 - 13.30	12.00 - 13.30
	Restaurant	18.00 - 20.00	Closed

Rl coffee lounge: 08.30 - 16.00 Monday to Friday, closed weekend.

6.5.2. Pubs

Blewbury	The Red Lion
Chilton	Rose & Crown
East Hendred	The Plough
	Wheatsheaf
East Ilsley	The Crown and Horns
	The Swan
Steventon	The Cherry Tree
	The Fox
Wantage	The Lamb
	The Swan
West Hendred	The Hare
West Ilsley	The Harrow

7. APPENDICES

7.1 TOSCA ***Parameters***

Moderator:	H_2O 300K (Poisoned with Gd at 2.5 cm)
Beam size at sample:	40 by 40 mm (h x w)
Beam height	1165 mm from underside of flange to centre of the beam
Detectors	130 ^{3}He proportional counters for inelastic scattering (2.5 mm effective thickness 150 mm effective length) 4 ^{3}He diffraction detectors in backscattering 1 incident beam monitor (scintillator)

Distances

Moderator to chopper	9.5 m

Moderator to sample	17.0 m
Sample to detectors	0.7 m

7.2 List of TOSCA ***Specific GENIE commands***

`B2A:==@TOSCA$DISK0:[TOSCA.COMMAND]b2a`

! Converts binary files into ASCII

`CON:==@TOSCA$DISK0:[TOSCA.COMMAND]CONVERT`

! Conversion of X-scale from meV to cm^{-1}

`DER:==@TOSCA$DISK0:[TOSCA.COMMAND]DERIVATIVE`

! Double derivative of a workspace

`GAUS:==@TOSCA$DISK0:[TOSCA.COMMAND]NGAUS`

! Gaussian fits prog (T.Perring)

`LOADB2A`

! Loads 3 column (x,y,e) ASCII file

`LPR:==@TOSCA$DISK0:[TOSCA.COMMAND]LASER_PLOTS.COM`

! Screen capture and print

`STR:==@TOSCA$DISK0:[TOSCA.COMMAND]STRETCH`

! Stretches/compresses the X scale by requested factor

`SMO:==@TOSCA$DISK0:[TOSCA.COMMAND]SMOOGEN`

! Smoothing using Savitzky-Golay method

`SPIKE:==@TOSCA$DISK0:[TOSCA.COMMAND]SPIKE`

! Spike removal

`COMP:==@TOSCA$DISK0:[TOSCA.COMMAND]COMP`

! Correction for misaligned sample

```
TP:==@TOSCA$DISK0:[TOSCA.COMMAND]TEMP_PLOT.COM

TPC:==@TOSCA$DISK0:[TOSCA.COMMAND]TEMP_PLOT_CURRENT.COM

! Temperature plotting of current or old data

ZERO:==@TOSCA$DISK0:[TOSCA.COMMAND]ZERO_ERRORS

! Sets all error values in a workspace to zero

MUPHIP:==@TOSCA$DISK0:[TOSCA.COMMAND]MUPHIP.COM
DENSIP:==@TOSCA$DISK0:[TOSCA.COMMAND]DENSIP.COM

DAMP:==@TOSCA$DISK0:[TOSCA.COMMAND]DAMP.COM

ERA:==@TOSCA$DISK0:[TOSCA.COMMAND]ERA.COM

FR4:==@TOSCA$DISK0:[TOSCA.COMMAND]FOREHACK3B.COM

! Conversion of time-of-flight to energy transfer

SR4:==@TOSCA$DISK0:[TOSCA.COMMAND]FOREHACKSUM3B.COM

! Co-addition and conversion of sequential time-of-flight files to energy transfer

QR4:==@TOSCA$DISK0:[TOSCA.COMMAND]FOREHACKQ3B.COM

! To look at the data being collected
```

7.3 Detector Tables

7.3.1 WIRING.DAT

The wiring table can be downloaded here.

7.3.2 SPECTRA.DAT

The spectra table can be downloaded here.

7.3.3 DETECTOR.DAT

The detector table can be downloaded here.

7.4 Detector Voltages

LeCroy Channel	**Voltage**	**Comment**
0	0	Not used
1	1650	Inelastic detectors
2	1650	Inelastic detectors
3	1650	Inelastic detectors
4	1650	Inelastic detectors
5	1650	Inelastic detectors
6	1650	Inelastic detectors
7	1650	Inelastic detectors
8	1650	Inelastic detectors
9	1650	Inelastic detectors
10	1650	Inelastic detectors
11	0	Not used
12	975	Monitor
13	850	Inelastic detectors
14	850	Inelastic detectors

Channel 15 onwards are not used.

Page updated:
Page URL: http://www.isis.rl.ac.uk/molecularSpectroscopy/tosca/toscamanual.htm